

# **A Systematic Method For Scaling Of Industrial Mixers**

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## **Abstract**

### **A Systematic Method For Scaling Of Industrial Mixers**

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Mixing is a common unit operation in the cosmetic industry and other industry's governed by Good Manufacturing Practices (GMPs). Most mixing operations in this industry occur in tanks with baffled, anchor agitators to provide bulk movement of high viscosity, Non-Newtonian emulsions. They also have a homogenizer to reduce the droplet size of the emulsion, which allows for changes in the fluid's rheology. It is important for companies to know how to transfer products from one tank to another during scale-up. Without a systematic scale-up method, companies lose time and money as more trial batches are needed. The scaling method in this paper relies on knowledge of the "working" viscosity of a fluid, which is the viscosity at a temperature and a shear rate, with an optimized process for a smaller tank. To scale to another tank, homogenizer shear rate and the number of turnovers are kept constant. The shear rate can be used to find the rotor speed of the homogenizer, which can then be used to calculate its flow rate at the working viscosity. The batch volume is divided into the flow rate to find the time for one turnover, then multiplied by the required turnovers for the optimized process to find the time. The agitator is scaled by keeping its power input to batch volume (P/V) ratio constant. Power can be calculated using a dimensionless power number, which depends on Reynolds number and the agitator geometry. The working viscosity is used to calculate the power number and power, then the power is divided by the batch volume. The agitator speed for the new tank is changed until the P/V ratio is equal to the smaller tank's ratio. Mixing time data was collected for fluids for which pH or hydrogen peroxide concentration could be easily tracked. The mixing times were converted to a dimensionless number by multiplying by the agitator speed. The power number was calculated for each sample on the largest tank from its working viscosity. A power equation was fitted to have dimensionless mixing time as a function of power number. The data from the additional tanks were used to support the equations developed to calculate power number. A right tailed t-test was run for each tank with a null hypothesis that the variation in the power number calculation was greater than the variation in the mixing time measurements. To support the equations, there needs to be 95% confidence that the variation comes from the mixing time measurements. The test rejected the null hypothesis for each tank, thus supporting the equations for power number. The mixing time collection also revealed that two of the systems studied are being mixed on average ten times more than required. Reducing these values to around the required time would save over \$30k annually. Overall, this scaling method will save time and money as new products are introduced.

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## Nomenclature

D	Agitator (Rotor) diameter	m
N	Agitator (Rotor) rotational speed	s <sup>-1</sup> or min <sup>-1</sup>
$\mu_b$	Bulk viscosity	Pa·s or cP
x	Constant for $C_\infty$ extrapolation	Dimensionless
C	Constant for laminar power number correlation	Dimensionless
k	Constants for flow number, power number, and $C_\infty$ equations	Dimensionless (For $C_\infty$ mol/L or pH)
$\rho$	Density	kg/L
n	Exponent for flow number and power number correlations	Dimensionless
T <sub>final</sub>	Final process temperature	K or °C
N <sub>q</sub>	Flow number	Dimensionless
f	Fouling Factor	m <sup>2</sup> K/W
T <sub>initial</sub>	Initial process temperature	K or °C
T <sub>jacket</sub>	Jacket fluid temperature	K or °C
A <sub>jacket</sub>	Jacket heat transfer area	m <sup>2</sup>
h <sub>jacket</sub>	Jacket side convective heat transfer coefficient	W/m <sup>2</sup> K
Nu	Nusselt Number	Dimensionless
U	Overall heat transfer coefficient	W/m <sup>2</sup> K
P	Power	kW
P <sub>0</sub>	Power number	Dimensionless
Pr	Prandtl Number	Dimensionless
C <sub>p</sub>	Process fluid heat capacity	J/kgK
k <sub>process</sub>	Process fluid thermal conductivity	W/mK
A <sub>process</sub>	Process heat transfer area	m <sup>2</sup>
h <sub>process</sub>	Process side convective heat transfer coefficient	W/m <sup>2</sup> K
V	Process volume	L
Re	Reynolds number	Dimensionless
X	Scale-up factor for agitators in the turbulent regime	Dimensionless
$\delta$	Shear gap	m
t	Time	min
A <sub>wall</sub>	Total wall area	m <sup>2</sup>
$\mu$	Viscosity (At specific temperature & shear)	Pa·s or cP
k <sub>wall</sub>	Wall thermal conductivity	W/mK
x <sub>wall</sub>	Wall thickness	m
$\mu_w$	Wall viscosity	Pa·s or cP

## Table Of Contents

Abstract.....	ii
Acknowledgements.....	iii
Nomenclature.....	iv
List Of Figures.....	vi
List Of Tables.....	vii
1. Introduction.....	1-3
1.1. Purpose.....	1
1.2. Processing.....	1
1.3. Mixing In The Cosmetic Industry.....	2-3
2. Theory.....	3-7
2.1. Agitation.....	3-4
2.2. Homogenization.....	4-6
2.3. Heat Transfer.....	6-7
3. Materials.....	7
4. Methods.....	7-12
4.1. Agitator Correlations.....	7-8
4.2. Homogenizer Correlations.....	8
4.3. Heat Transfer Correlations.....	8-9
4.4. Mixing Time Measurements.....	9-10
4.5. Working Viscosity.....	10
4.6. Mixing Time Correlation And Validation.....	11-12
5. Results And Discussion.....	12-18
5.1. Agitation Correlations.....	12-13
5.2. Homogenization Correlations.....	13-15
5.3. Heat Transfer Correlations.....	15-16
5.4. Mixing Time Measurements.....	16
5.5. Working Viscosity.....	17
5.6. Mixing Time Correlation And Validation.....	17-19
6. Impact And Conclusion.....	19
7. References.....	20
8. Appendices.....	21-43
8.1. Appendix A.....	21-26
8.2. Appendix B.....	27-30
8.3. Appendix C.....	31-36
8.4. Appendix D.....	37-39
8.5. Appendix E.....	40-42
8.6. Appendix F.....	43
9. Curriculum Vitale.....	44-46

## List Of Figures

Figure 1:	Laminar mixing agitators.....	2
Figure 2:	Anchor agitator with motor, stationary baffles, and moving paddles.....	2
Figure 3:	Graph of $Re$ versus $P_o$ and the equation relationships in each flow regime.....	4
Figure 4:	Shear gap (red) between the rotor (inner diameter) and stator (outer diameter)....	5
Figure 5:	Flow number versus Reynolds number.....	6
Figure 6:	Agitator speed conversion between tanks at 10,000 cP and full capacity.....	12
Figure 7:	Agitator speed conversion between tanks at 50,000 cP and full capacity.....	13
Figure 8:	Shear rates at different rotor speeds for the tank homogenizers.....	14
Figure 9:	Homogenizer flow rate at different speeds and viscosities for V-301.....	15
Figure 10:	Cooling curve for tank V-401 at 40 RPM and a final temperature of 25°C.....	16
Figure 11:	Viscosity of the Kenra Developer at different temperatures and shears ( $n=3$ )....	17
Figure 12:	Mixing time curve at different viscosities and rotational speeds for V-301.....	18
Figure 13:	Mixing time data graphed against the confidence intervals of the correlation....	19

## List Of Tables

Table 1:	Table of scale factors for agitation based on intended results.....	3
Table 2:	Parameters for heat transfer correlations, independent of tank system.....	9
Table 3:	Products tested for mixing time, with test, and time samples were taken.....	9
Table 4:	Brookfield viscometer settings for different shear rates.....	10
Table 5:	Constants for the agitator power number correlations.....	12
Table 6:	Tank area and fouling coefficients for heat transfer correlations.....	15
Table 7:	Average mixing time for each product.....	16

## **Introduction**

Mixing is a common unit operation in the cosmetic industry and other industry's governed by Good Manufacturing Practices (GMPs). Mixing occurs within a tank, facilitated by a variety of types of agitators. Homogenization normally occurs via a rotor-stator device that can either be mounted at the bottom of the tank or in a pipe connected to the tank. There are agitation scaling rules in literature, but most assume turbulent conditions and need to be adapted for use with high viscosity products. However, homogenization scaling is less studied due to the complexity and variety of the flow patterns within the devices.

### **1.1. Purpose**

The purpose of this project was to create a simple, systematic way to scale between six production mixing tanks and one pilot tank at the C-Care facility. The scaling rules created provides an empirical basis to reduce the time it takes to introduce products to a new tank.

### **1.2. Chemistry And Processing**

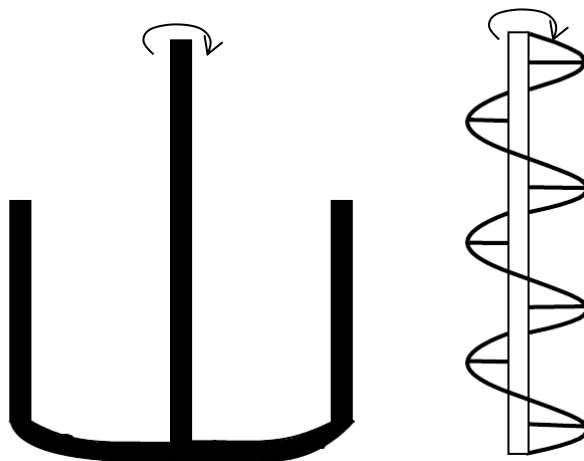
The cosmetic industry has a variety of products; including, shampoos, conditioners, hair colors, hair sprays, shave butters, hair developers, shave creams, and hair gels. Most of these products are an emulsion except for some water and polymer-based products. Emulsions can either be oil suspended in water or water suspended in oil.[1] Cosmetic emulsions are mainly characterized by an oil-phase suspended in a water-phase with the addition of an emulsifier. The emulsifier contains both a hydrophilic and lipophilic component that helps to stabilize the emulsion. The amount of each phase can be used to determine the hydrophilic-lipophilic balance (HLB) of the system. HLB is determined by the ratio of the molecular weight of the hydrophilic to hydrophobic parts of the molecule. An emulsifier that has a similar HLB value to the system adds stability to the emulsion.[1] Since most cosmetic products are oil in water emulsions, most emulsifiers used have HLB values greater than ten, meaning they are more hydrophilic. A common stabilizer of the oil-phase in cosmetics is hexadecanol which requires an emulsifier with an HLB value of 15 to balance the hydrophobic effects of hexadecanol.

The long-term stability of the emulsion depends on droplet size, as droplet size decreases coalescence and gravitational separation tends to decrease. This is because the area to volume ratio of the droplets increases which leads to more ionic interactions between the droplets, countering the gravitational forces. The droplet size also plays a role in the rheological and optical properties of the emulsion.[2] This requires the use of high intensity mixing to create enough shear to break down the droplets to the desired size. This is accomplished by using a rotor-stator homogenizer. A low shear agitator is also used to circulate the product within the tank to create a homogenous emulsion.



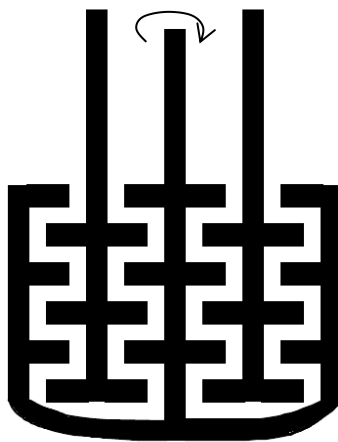
### 1.3. Mixing In The Cosmetic Industry

Cosmetic mixing occurs in batch processes with a sweeping agitator and a homogenizer attached. Since products can reach viscosities greater than 200,000 cP anchor or helical ribbon agitators (Figure 1) are used for mixing.



**Figure 1.** Laminar mixing agitators. Right: Helical Ribbon, Left: Anchor

All the agitators used in this study are modified anchor agitators. These are anchors with fixed baffles and moving paddles. An example of this type is shown in Figure 2, where the middle shaft is the agitator shaft and the others are baffles that are fixed in place.



**Figure 2.** Anchor agitator with motor, stationary baffles, and moving paddles.

To reduce the droplet size of the oil-phase, rotor-stator homogenizers are used to increase the shear stress on the droplet. Many different stator geometries can be used for homogenization depending on the rheological properties of the emulsion involved. New homogenizers also can have a moving stator to provide more shear stress to the product. These homogenizers can operate with a stationary, co-directional, or counter-directional

stator respective to the motion of the rotor. This makes creating scale-up rules for homogenizers difficult as there are many operational configurations with respect to speed, moving stator, and stator geometry.

## 2. Theory

### 2.1. Agitation

Most scale-up methods are based from geometric similarity of the mixing vessels and turbulent conditions. Equation 1 is then used with a scale factor  $X$  to relate the agitator speeds  $N$  to the diameters  $D$ , depending on what property is to be kept constant (Table 1).[3]

$$ND^X = Constant \quad (1)$$

**Table 1.** Table of scale factors for agitation based on intended results.[3]

Scale Factor (X)	Rule/Result
1.0	Constant tip speed, maximum shear
0.85	Off-bottom solids suspension
0.75	Conditions for average suspension
0.67	Constant P/V, constant mass transfer rate
0.5	Constant Reynolds number, similar heat transfer
0.0	Constant speed, equal mixing time

In cosmetics, engineers are rarely concerned with suspending solids into the emulsion as the primary concern is getting the liquid components homogenous. When using constant speed, the scaled-up batch will eventually reach a volume where the larger agitator will draw more power than the motor can output. This makes scaling with respect to constant speed not feasible or at least very expensive as a high capacity motor is needed.

Tip speed, velocity tangent to the end of the agitator, can be easily kept constant across tanks but is only valid under turbulent conditions. Unfortunately, cosmetic mixing rarely has turbulent flow because of the high viscosity of the emulsions. The above equation describes the P/V ratio in terms of the turbulent regime by cancelling out factors that are constant in the turbulent regime and assuming the volume is proportional to the cubic tank diameter. It can be used without these assumptions by calculating the power exerted by the motor then dividing by the batch volume. Power can be calculated using Equation 2, where  $P_O$  (power number) is an empirical constant which changes with the specific agitator geometry and is dimensionless.[4]

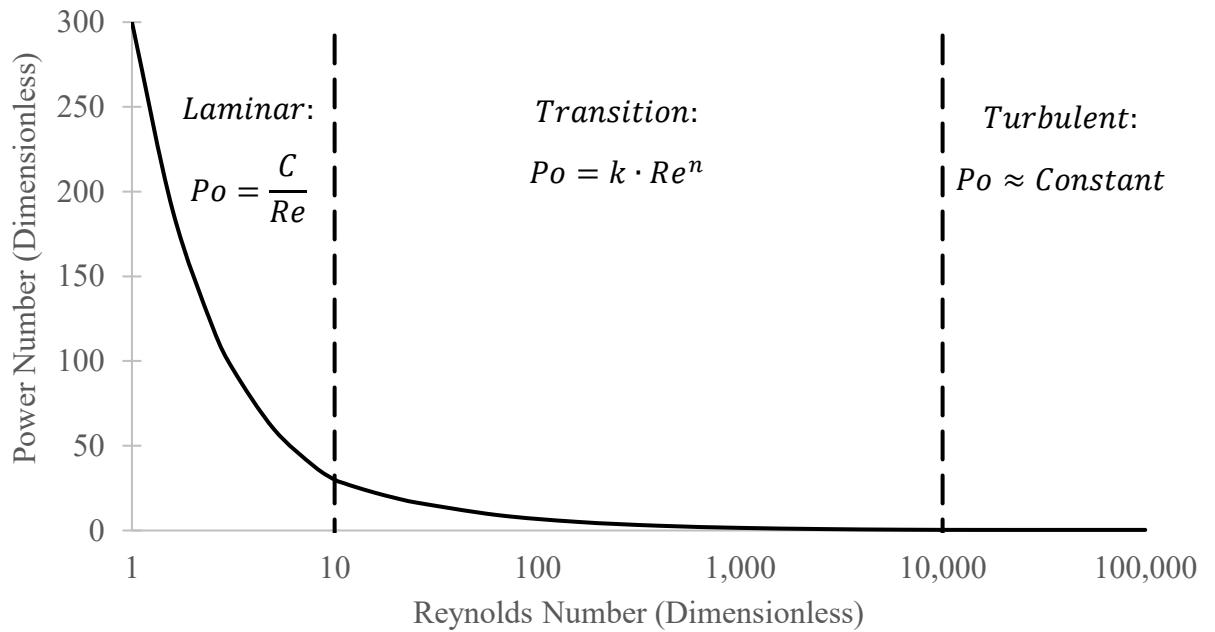
$$P = \rho P_O N^3 D^5 \quad (2)$$

Agitator speed  $N$ , density  $\rho$ , and agitator diameter  $D$  are all specified when trying to calculate the power for an agitator with a known process. Power number is a function of

the agitator geometry and Reynolds number, see Figure 3.[5]–[7] Reynolds number can be calculated from Equation 3.

$$Re = \frac{\rho ND^2}{\mu} \quad (3)$$

The viscosity  $\mu$  used in this formula is the “working viscosity”, which means the viscosity at the process temperature and at the shear rate of the agitator. The transition to turbulent flow occurs at  $Re = 10,000$  and to laminar at  $Re = 10$ . [7] It has been shown that power number is approximately constant in the turbulent regime, inversely proportional to Reynolds number in the laminar regime, and has a power relationship with Reynolds number in the transition regime (Figure 3).[6], [8]–[10]



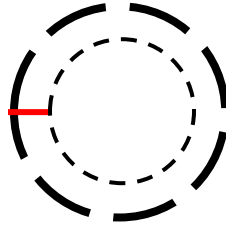
**Figure 3.** Graph of  $Re$  versus  $P_o$  and the equation relationships in each flow regime.

This curve can be constructed for different types of agitators using the three equations above with a conditional statement to describe the transition between turbulent ( $Re \geq 10,000$ ) and laminar ( $Re \leq 10$ ). Power number in the turbulent regime is approximated as constant because the changes are so small relative to changes in the transition and laminar regime. This makes calculations in industry simple when trying to calculate the power required of an agitator in the turbulent regime. Once the power number and power of the agitator is known then the  $P/V$  ratio can be calculated by dividing the power  $P$  by the batch volume  $V$ .

## 2.2. Homogenization

Rotor-stator homogenizers are widely used in the production of cosmetic products to reduce the droplet size of an emulsion to change the rheology of the product. However, there is little scientific literature on direct scaling of homogenizers due to the variety of operational configurations.[11], [12] Homogenizers can either be scaled using constant tip speed, shear rate, P/V ratio, or number of tank turnovers. Tank turnovers are defined as the number of times a batch is completely turned over in a certain amount of time. Shear rate is calculated using Equation 4, the rotor diameter  $D$ , and the rotor speed  $N$ . Where  $\delta$  is the radial clearance, or shear gap, between the rotor and stator (Figure 4). The P/V ratio can be calculated the same way as an agitator by defining a power number relationship, calculating the power, and dividing by the batch volume. However, the power number is not easily defined for homogenizers due to not having a simple way to measure the power of the rotor.

$$\gamma = \frac{\pi DN}{\delta} \quad (4)$$

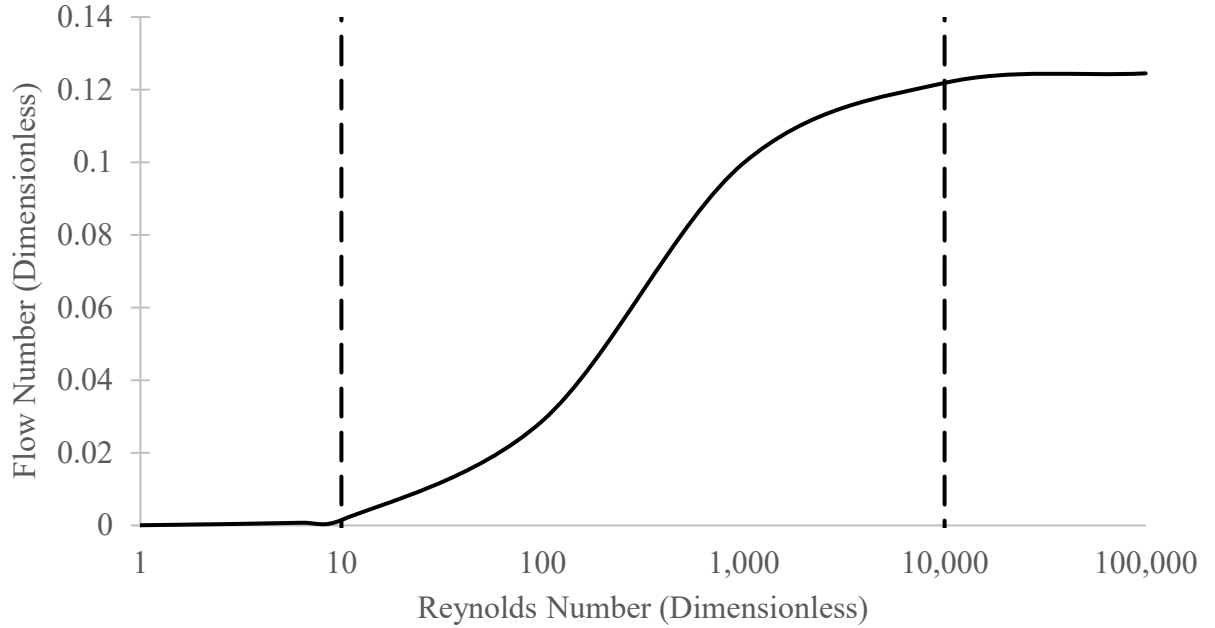


**Figure 4.** Shear gap (red) between the rotor (inner radius) and stator (outer radius).

Turnovers can be calculated using the flow rate  $Q$  of the homogenizer and the batch volume. The flow rate can be found by measuring values from a downstream flow meter or by theoretically calculating the flow rates. As the power of an agitator was calculated, the flow rate through a homogenizer can be related to viscosity through dimensionless numbers. The flow rate is calculated from Equation 5, where  $N_Q$  is a dimensionless flow number, which is an empirical number that relates the flow rate to the homogenizer geometry and flow regime. For a homogenizer,  $N$  is the rotor speed and  $D$  is the rotor diameter.[13]

$$Q = N_Q N D^3 \quad (5)$$

The flow number can be related to Reynolds number, where it's constant in the turbulent and laminar regime.[13], [14] An example curve is shown in Figure 5 and takes a “S” shape.



**Figure 5.** Flow number versus Reynolds number.

This curve takes the form of the Equation 6, where  $N_Q$  is the flow number,  $N_{Qmax}$  is the maximum flow number,  $n$  &  $K$  are constants dependent on the homogenizer, and  $Re$  is Reynolds number. The maximum flow number can be calculated using the maximum flow rate of water (specified by the vendor) and Reynolds number, then solving for the flow number in Equation 5.

$$N_Q = N_{Qmax} \left( \frac{Re}{Re+K} \right)^n \quad (6)$$

Finally, the flow rate can be used to calculate the number of turnovers by multiplying the flow rate by the homogenization time and dividing by the volume of the batch.

### 2.3. Heat Transfer

Heat transfer for a jacketed vessel is calculated using the differential equation for heat transfer with an overall heat transfer coefficient  $U$  (Equation 7).[15]

$$\frac{dT}{dt} = \frac{UA_{process}}{\rho V C_p} (T_{jacket} - T) \quad (7)$$

Where  $T$  is the temperature,  $A_{process}$  is the process side area,  $\rho$  is the process fluid density,  $V$  is the batch volume,  $C_p$  is the process fluid heat capacity, and  $T_{jacket}$  is the jacket fluid temperature.

The variables in the equation are defined by the process besides the overall heat transfer coefficient which is calculated using Equation 8.[16]

$$\frac{1}{U} = \frac{1}{h_{jacket}} \left( \frac{A_{process}}{A_{jacket}} \right) + \frac{x_{wall}}{k_{wall}} \frac{A_{process}}{A_{wall}} + f + \frac{1}{h_{process}} \quad (8)$$

Where  $A$  heat transfer area of the indicated region,  $h$  is the convective heat transfer coefficient of the region,  $k_{wall}$  is the wall thermal conductivity,  $f$  is the fouling factor, and  $x_{wall}$  is the wall thickness.

The overall heat transfer coefficient varies with changes in surface area of the vessel and the process side heat transfer coefficient  $h_{process}$  which can be calculated from the Nusselt number  $Nu$  (Equation 9).[16]

$$Nu = 0.69 Re^{\frac{1}{2}} (Pr.)^{\frac{1}{3}} \mu_R^{0.14} = \frac{h_{process}}{k_{process}} D \quad (9)$$

The Nusselt Number is defined by the Reynolds number (Equation 10), Prandtl number  $Pr$  (Equation 11), and viscosity ratio  $\mu_R$  (Equation 12). The process side heat transfer coefficient can then be calculated using the agitator diameter  $D$  and the process fluid thermal conductivity  $k_{process}$ .

$$Re = \frac{\rho N D^2}{\mu_b} \quad (10)$$

$$Pr = \frac{\mu_b C_p}{k_{process}} \quad (11)$$

$$\mu_R = \frac{\mu_w}{\mu_b} \quad (12)$$

Where  $\mu_b$  is the bulk process fluid viscosity and  $\mu_w$  is the viscosity at the wall.

Once the parameters have been defined then the differential equation (Equation 7) can be solved for time.

### 3. Materials

Hoyu Acid Hair Color, Hoyu Alkaline Hair Color, Kenra Direct Dye Hair Color, Dr. Carver's Shave Butter, Kenra Developer.

### 4. Method

Correlations were developed for the agitator speed, homogenizer speed, homogenizer flow rate, and heat transfer separately. Data was collected to determine the mixing time and working viscosity.

#### 4.1. Agitator Correlations

The agitators were scaled using a constant P/V ratio. This allows the agitator speed to become a function of both viscosity and batch volume. For the two largest tanks, the

relationship between Reynolds and power number was established experimentally by the vendor. To calculate the other agitator's turbulent regime power number, the tip speeds were set equal and the power number was changed to keep the P/V ratio (volume at maximum capacity) constant across all the mixers. This was done using Excel Solver to minimize the sum of least squares (SLS) between the P/V ratio of the pilot and other tanks.

To find the laminar constant  $C$  for the other mixers the ratio between  $C$  and the turbulent regime power number for the large tanks were multiplied by the turbulent power number for the others.

Then the transition regime constants ( $k$  &  $n$ ) were found by minimizing the SLS of the difference between the transition Reynolds number (10 and 10,000) and a tenth into the transition region (10.1 and 9,999.9). Once these constants were established, Excel Solver was used to change the speed of the agitators to set the P/V ratio equal to the pilot agitator. The constraints of the Solver were set to keep the speed within the vendor specified ranges of the agitators and the power under 80% of the motor capacity. Finally, the tank speeds were graphed against the pilot speeds at different viscosities and fractional volume capacities.

#### **4.2. Homogenizer Correlations**

Since homogenizers are used to reduce droplet size, which depends on the shear rate, constant shear rate was used for scale-up. The number of turnovers was also kept constant to determine the length of time to run the homogenizer.

Shear rate was calculated using Equation 4 and was used to set the speed of the rotor for the scaled homogenizer. To find the flow number in the turbulent regime, the maximum (turbulent) flow number was calculated using Equation 5 at the maximum flow rate and speed for the homogenizer. The minimum (laminar) flow number was calculated by taking 45% of the maximum flow number, which is the percent difference reported in literature.[14] The constants in Equation 6 were found using Excel Solver by minimizing the SLS between the calculated transitional flow numbers and the ones established above at  $Re=10$  and  $Re=10,000$ . The flow rate was then calculated through the homogenizer at a certain viscosity. Then, the time for one turnover was calculated by dividing the tank volume by the flow rate. Finally, this time was multiplied by the number of turnovers needed in the other tank to find the homogenization time.

#### **4.3. Heat Transfer Correlations**

The heat transfer equations were used to estimate the amount of time it takes to cool or heat a batch. Once Equation 7 was integrated and solved for time, the overall heat transfer coefficient was calculated using the parameters in Table 2 as well as the equations in the theory section. Jacket fluid temperatures used were 95°C for hot water, 186°C for steam, and 4°C for chilled water.

**Table 2.** Parameters for heat transfer correlations, independent of tank system.

Parameter	Value
$h_{\text{jacket}}$	Hot water: 5, Steam: 10, Chilled water: 1 kW/m <sup>2</sup> K
$k_{\text{process}}$	0.6 W/mK
$k_{\text{wall}}$	17 W/mK
$C_p$	4.18 J/kgK (Carbopol 0.5%, 4.5 kJ/kgK)

The results were then plotted on graphs with time on the y-axis versus the temperature to cool or heat to on the x-axis. This was done for ease of use in industry, as the time it takes to cool to a certain temperature is more important than knowing the temperature after cooling for a certain amount of time.

#### 4.4. Mixing Time Measurements

Mixing time measurements were taken on products that had a specified mixing step after the addition of an acid or hydrogen peroxide. Products with an acid addition were tested for pH changes using a Thermo Scientific A112 pH meter and hydrogen peroxide changes were tested via manual titrations using a Mettler Toledo Titrator T50. These products are listed in the material section and data collection was done with the help of the production team. Samples were pulled from the top of the tank since the company had previously proven that there was not stratification. A sample was pulled prior to the addition of the chemical to have a baseline measurement. Then five to six samples were pulled at certain time points during the mixing step depending on the duration of the mixing step, as listed in Table 3.

**Table 3.** Products tested for mixing time, with test, and time samples were taken.

Product	Test	Time Increments
Kenra Developer	H <sub>2</sub> O <sub>2</sub> Concentration	0, 5, 10, 15, 20 min
Hoyu Acid	pH	0, 10, 15, 20, 25, 30 min
Hoyu Alkaline	pH	0, 2, 3, 4, 5 min
Kenra Direct Dye	pH	0, 2, 3, 4, 5 min
Dr. Carver	pH	0, 10, 15, 20, 25, 30 min
Thayer's Toners	pH	0, 10, 15, 20, 25, 30 min

Once the data at the time intervals was recorded it was normalized using Equation 13.

$$Normalized = \left| 1 - \frac{C_i - C_0}{C_\infty - C_0} \right| \quad (13)$$

Where  $C_0$  is the measured value (pH or H<sub>2</sub>O<sub>2</sub> concentration) prior to the addition of the acid or peroxide. This value either measured or estimated using an average difference between  $C_0$  and  $C_\infty$  for the product from those that were measured.  $C_\infty$  is the value at time infinity, meaning complete mixing.  $C_\infty$  was taken for the Kenra Developer's, Dr.



Carver's, and Thayer's products as the value on the certificate of analysis (Quality Assurance record sheet), as this was the last chemical addition prior to discharge. The other products had to be estimated using an extrapolation of Equation 14 since the chemicals were added in the middle of the batch.

$$C_i = kx^{1/t} \quad (14)$$

This equation was fitted to the data by changing the constants ( $k$  &  $x$ ) using the SLS between the measured values at the time points  $t$  and the estimated values from the equation  $C_i$ . Then the limit was taken at time goes to infinity, which was determined to be the constant  $k$ . This constant was then used as the value for  $C_\infty$  for those products.

The equation for normalization shows how the parameter varies over time at a specific sampling point. If there was no absolute value then a curve of the sampled measurements over time would be a sinusoidal that goes to zero, meaning no variations in the measurements. A value of one would mean that the chemical has yet to be mixed in and that point is still at  $C_0$ . Taking the absolute values allows the normalized data to be fitted to an exponential decay model. This can then be solved for time when the model becomes 0.05. This is the time where the variations reach within  $\pm 5\%$  of  $C_\infty$ , thus implying 95% homogeneity. Outliers were removed using at 1.5 times the interquartile range to create a low and a high range.

#### 4.5. Working Viscosity Measurements

To determine the viscosity to use in the above equations, the viscosities of the products listed in Table 4 were analyzed over different temperatures (25, 35, 45, 50, 65, 70°C) and shear rates (low, medium, high) using a Brookfield Digital D+ Viscometer. The medium shear rate was defined as the shear rate of the spindle and at the speed used to determine if the final product is in specification, according to the certificate of analysis. Then to find the low and high shear, the speed was adjusted as high or low as possible without going out of range of the viscometer. Table 4 lists the spindles and speeds used to analyze each product at each temperature. Each measurement was taken in triplicate and the average  $\pm$  standard error was recorded.

**Table 4.** Brookfield viscometer settings for different shear rates.

Product	Spindle	Low	Medium	High
Hoyu Acid	RVT-3	2.5 RPM	5 RPM	10 RPM
Hoyu Alkaline	RVT-3	5 RPM	10 RPM	20 RPM
Kenra (DD) Semi-Permanent	RVT-C	2.5 RPM	5 RPM	10 RPM
Dr. Carver's Shave Butter	LVT-F	3 RPM	6 RPM	12 RPM
Kenra Developer	RVT-2	2.5 RPM	5 RPM	10 RPM

#### 4.6. Mixing Time Correlation And Model Validation

The mixing time data was used to create a correlation between dimensionless mixing time and power number. Dimensionless mixing time is the product of the mixing time measured from production and the respective agitator speed. Since power number and dimensionless mixing time varies with Reynolds number in a power relationship, these relationship between these two numbers also follows a power relationship.

The data from the largest tank (V-301) was used to establish this correlation since the second largest did not have any products with defined mixing steps. For each sample that was measured over time, the viscosity of the time zero sample was also measured using the medium shear setting. The viscosity versus temperature data from the working viscosity samples was normalized by dividing the high shear data by the medium shear value at 25°C. Then this data was fitted with a second-order polynomial to create an equation for a viscosity ratio of a product at a specific temperature. This ratio was then multiplied by the viscosity recorded from samples collected for each point during the mixing time data collection. This was used to find the working viscosity for the product at temperature in the process for the specific sample.

This viscosity was used to find the power number for each of the data points using the power number equations and respective constants. The data was averaged for each product category on V-301 and the mean dimensionless mixing time was graphed against the mean power number. Then a power equation was fitted to the points on the graph to have an equation for dimensionless mixing time versus power number. By plotting points representative of the 70% and 95% confidence intervals for the averages, curves were created for these intervals as well.

To validate the constants used in the power number correlations, mixing time data from the three smaller tanks (V-401, V-411, and V-437) were used in a right tailed t-test. The dimensionless mixing time for each sample was estimated using calculated power number equation created from V-301 data. The equation for the 95% confidence interval was also used to find estimated points for each power number. The average percent difference, across all tanks, between the estimated dimensionless mixing time and its 95% confidence interval was used as the population mean. The sample mean was calculated for each tank as the average percent difference between the estimated and measured dimensionless mixing time value. The population mean describes the variation in the measurements used to establish the power number to dimensionless mixing time equation. The sample mean describes the variation in those measurements and the variations in the equations used to calculate power number. If there was no variation both these means would be zero. The null hypothesis for this system is that the sample mean is greater than the population mean. Thus, the alternative hypothesis is the sample mean is less than or equal to the population mean. Rejecting the null hypothesis means that there is 95% confidence that the variation comes from the measurements for mixing time rather than the power number equation.

The t-statistic was calculated as the difference between population and sample mean divided by the standard error of the sample. Excel was used to calculate the p-value of a right tailed t-test using the t-statistic for each tank.

## 5. Results And Discussion

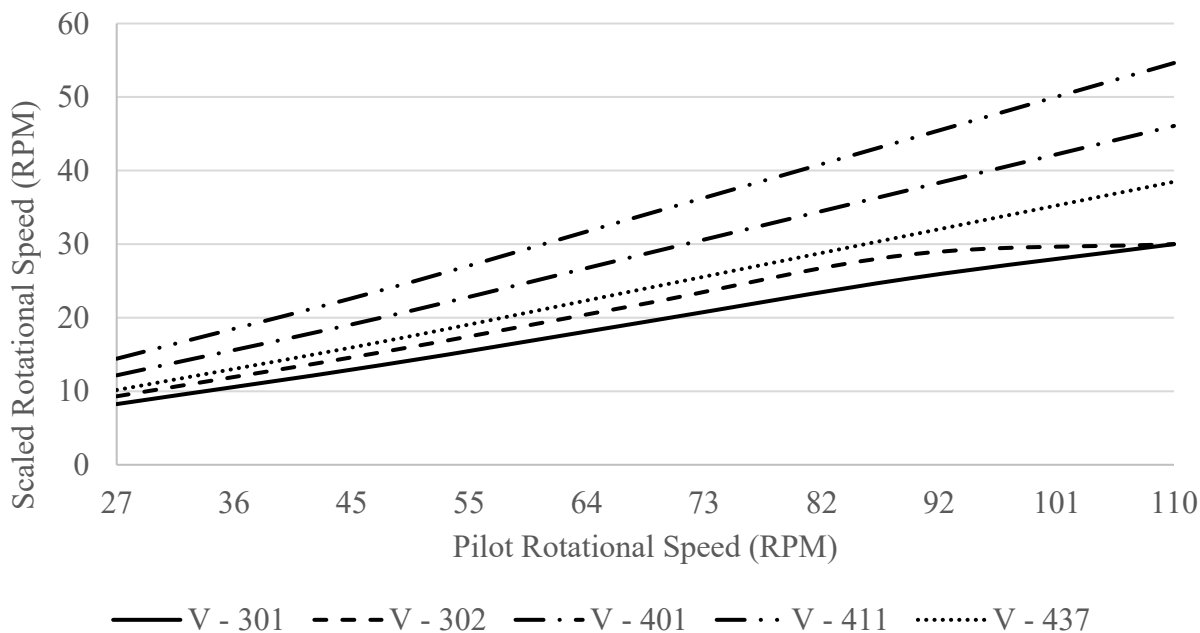
### 5.1. Agitation

Using the method described, the constants from the equations in Figure 3 were evaluated. Table 5 shows the three constants for each tank, where V-301 and V-302 are the two largest tanks with the established correlation provided by the vendor.

**Table 5.** Constants for the agitator power number correlations.

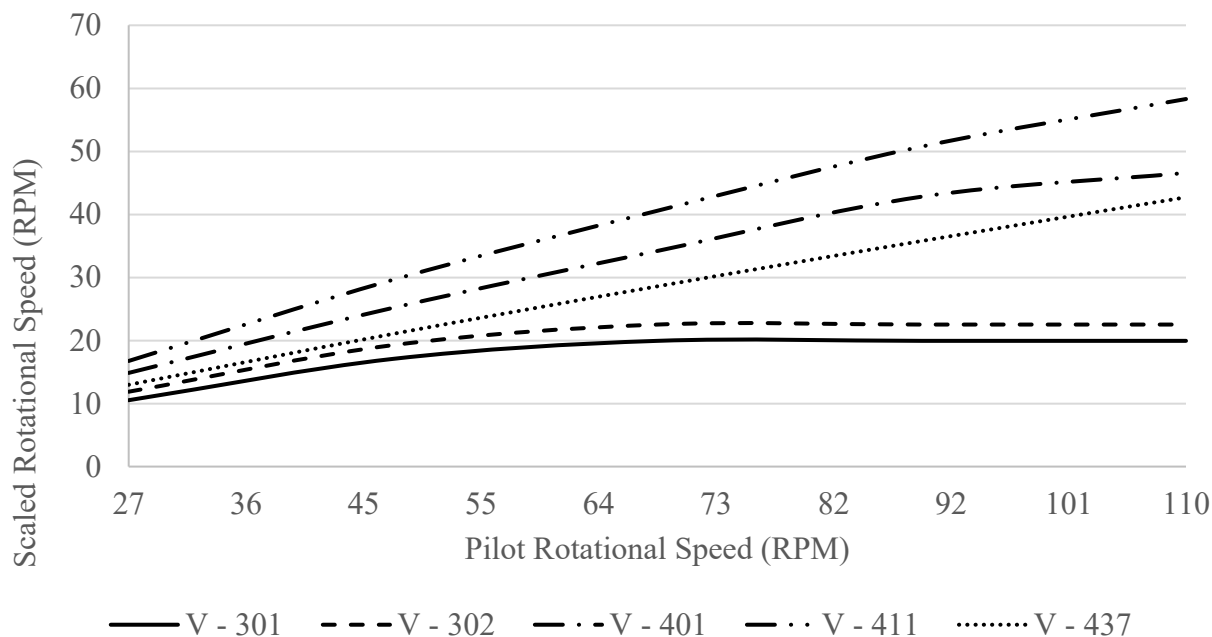
Tank	Capacity	Laminar Constant (C)	Transition Constant (k)	Transition Constant (n)	Turbulent Po
V-301	8,000 L	300	131.2	-0.64	0.35
V-302	6,000 L	300	131.2	-0.64	0.35
V-401	1,000 L	114	49.9	-0.64	0.13
V-411	600 L	106	46.4	-0.64	0.12
V-437	2,000 L	141	61.6	-0.64	0.17
V-421	30 L	38	16.6	-0.64	0.04

Graphs were then created keeping the P/V ratio constant to scale the agitator speed of the pilot scale mixer (V-421) to the larger tanks. An example graph for a product with a viscosity of 10,000 cP and full capacity tanks is shown in Figure 6.



**Figure 6.** Agitator speed conversion between tanks at 10,000 cP and full capacity.

The line on V-302 begins to become constant towards the end of the x-axis because it reaches the maximum RPM of the agitator. At higher viscosities the lines become constant because the power input needed reaches the limit imposed of 80% of the motors capacity. These limits are shown in Appendix F for each tank. An example of this is shown in Figure 7, where the larger tanks begin to reach a constant speed due to the high viscosity.

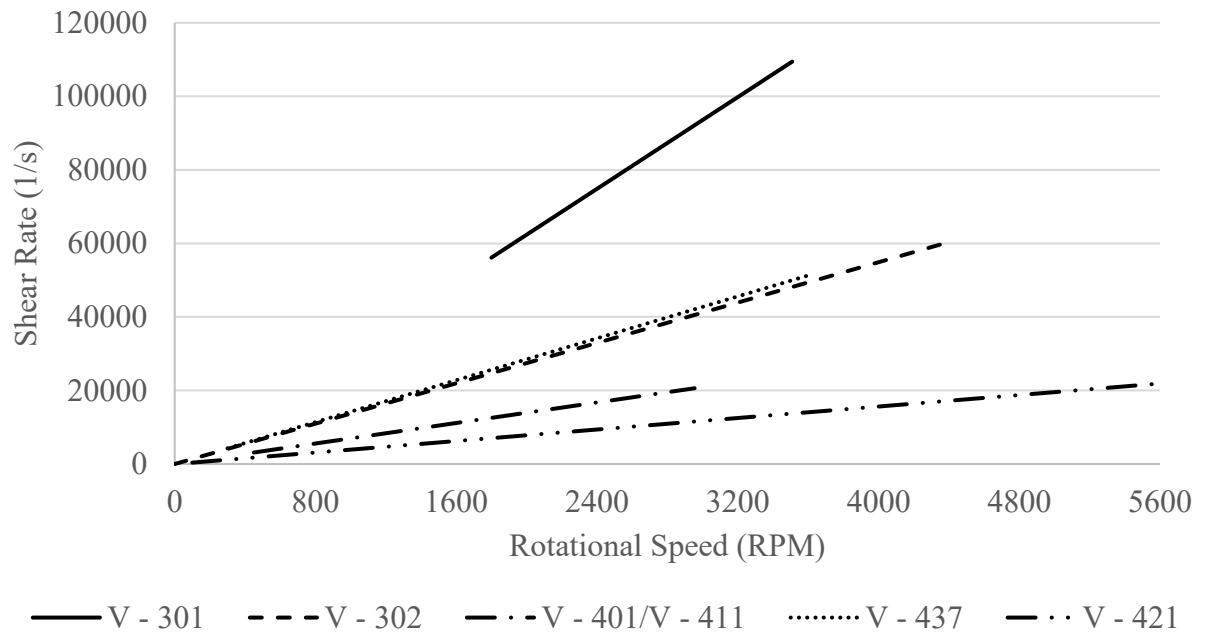


**Figure 7.** Agitator speed conversion between tanks at 50,000 cP and full capacity.

Graphs at for viscosities at 1, 100, 1,000, and 100,000 cP are shown in Appendix A.

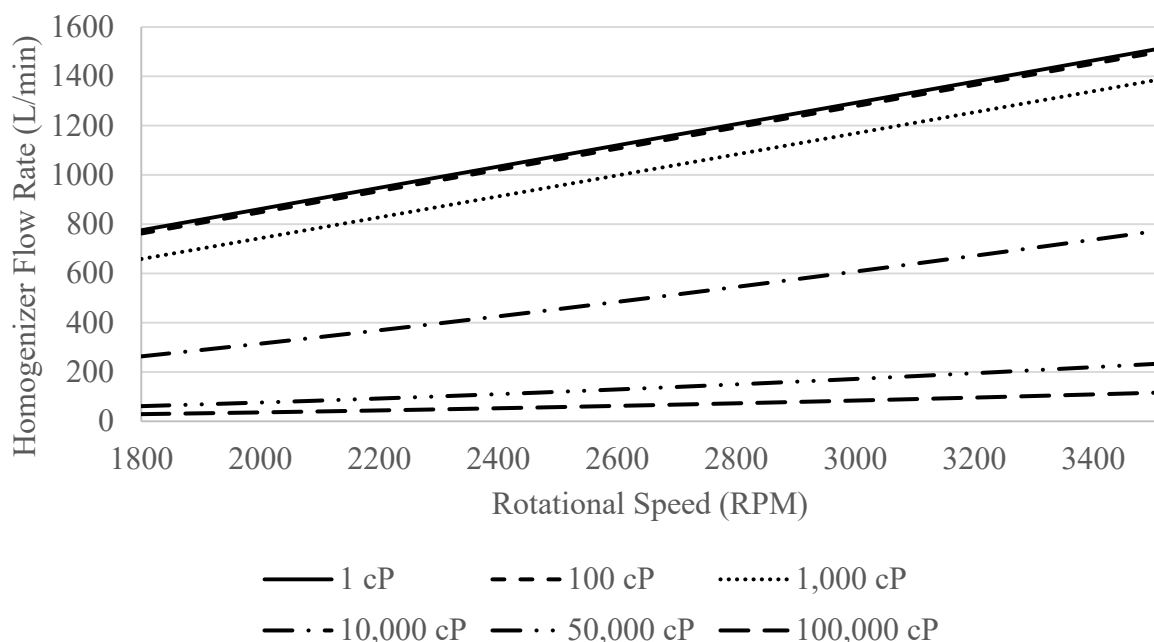
## 5.2. Homogenizer Correlations

The shear rates for each tank is shown in Figure 8. Since the shear gaps between each of the homogenizers are not similar, the resulting shear rates are different. This makes it difficult to set the shears equal especially when scaling-up from the pilot mixer.



**Figure 8.** Shear rates at different rotor speeds for the tank homogenizers.

Graphs were also made for the flow rate through each homogenizer at different rotor speeds and viscosities. Figure 9 shows an example graph for the largest tank (V-301). The constants for the flow number equation (Equation 6) were 100 for  $k$  and 1.2 for  $n$  for all tanks.



**Figure 9.** Homogenizer flow rate at different speeds and viscosities for V-301.

Graphs for the flow rates through the rest of the homogenizers are shown in Appendix B.

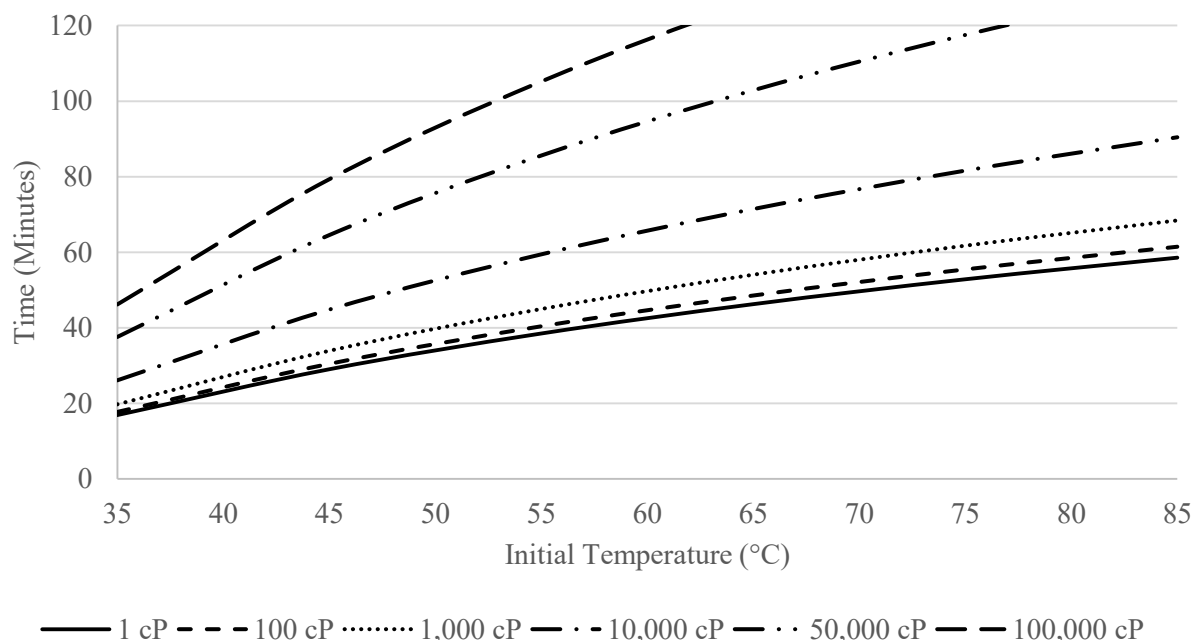
### 5.3. Heat Transfer Correlations

The required areas and fouling factors were calculated from equipment diagrams and from temperature probe data from the tanks (Table 6).

**Table 6.** Tank area and fouling coefficients for heat transfer correlations.

Tank	Area Process Side	Area Jacket Side	Area Wall	Fouling
V-301	18.57 m <sup>2</sup>	25.12 m <sup>2</sup>	26.74 m <sup>2</sup>	0.037
V-302	15.75 m <sup>2</sup>	19.95 m <sup>2</sup>	20.78 m <sup>2</sup>	0.039
V-401	4.44 m <sup>2</sup>	6.68 m <sup>2</sup>	6.49 m <sup>2</sup>	0.04
V-411	3.20 m <sup>2</sup>	4.21 m <sup>2</sup>	4.08 m <sup>2</sup>	0.04
V-437	7.03 m <sup>2</sup>	7.26 m <sup>2</sup>	7.67 m <sup>2</sup>	0.05
V-421	0.43 m <sup>2</sup>	0.52 m <sup>2</sup>	0.64 m <sup>2</sup>	0.04

Curves (Figure 10) were then made for heating and cooling of each tank at an agitator speed in the middle of the range. All the charts were made with 25°C as the final cooling temperature or initial heating temperature because this is the temperature that the tanks are discharged at. Time was put on the y-axis with temperature on the x-axis, which is inverse of the way the variables are related in the equation. This is for ease of use, as an engineer would want to know the estimated time to cool from one temperature to another rather than the temperature it will be after a certain amount of time.



**Figure 10.** Cooling curve for tank V-401 at 40 RPM and a final temperature of 25°C.

Additional heating and cooling curves for the rest of the tanks are listed in Appendix C.

#### 5.4. Mixing Time

Mixing time was measured at least three times per product then averaged. The averages and standard deviations are shown in Table 7.

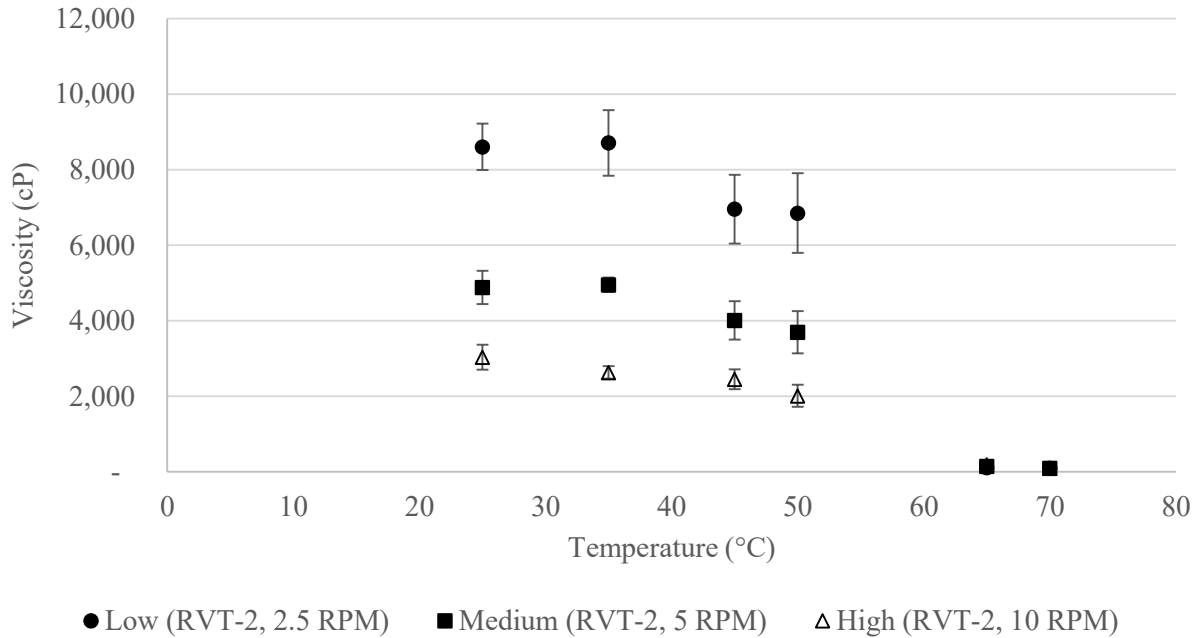
**Table 7.** Average mixing time for each product.

Product	Mixing Time
Dr. Carver's Shave Butter	$30.8 \pm 13.0$ minutes
Hoyu Acid	$2.2 \pm 1.2$ minutes
Hoyu Alkaline	$8.7 \pm 2.9$ minutes
Kenra Developer	$21.4 \pm 7.4$ minutes
Kenra (DD) Semi Permanent	$15.4 \pm 7.6$ minutes
Thayer's Toner	$4.0 \pm 3.3$ minutes

For most of the products the specified mixing step in the process is appropriate according to these measurements. However, the Thayer's and Hoyu Acid products are being overmixed. Currently, the mixing time for Hoyu Acid is 30 minutes, which is 13 times more than needed. Depending on the type of Thayer's Toner, the mixing step is either 30 or 45 minutes, which is greater than 7 times more than needed. If these steps were to be reduced to 10 minutes for Hoyu and 20 minutes for Thayer's, then the company would save \$30-40k annually.

### 5.5. Working Viscosity

Viscosities of each product were graphed at different temperatures and shear rates (Figure 11). Since the products are emulsions the viscosities vary differently than the normal exponential decay relationship. This is because the 1-hexadecanol used in the emulsion stabilizes the viscosity until it the alcohol melts around 55°C.[17] Once it melts it becomes liquid and drops to a low, Newtonian viscosity since the emulsion is around 80% water.



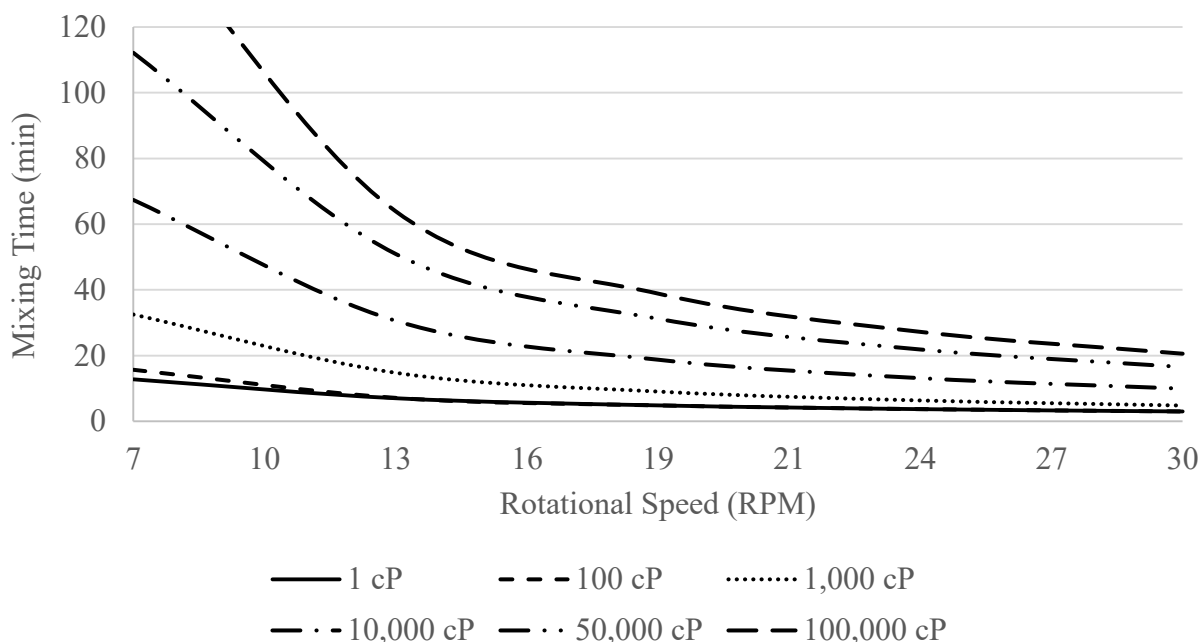
**Figure 11.** Viscosity of the Kenra Developer at different temperatures and shears (n=3).

Additional working viscosity curves for Kenra Semi Permanent Hair Colors, Dr. Carvers Shave Butter, Hoyu Hair Colors, and Thayer's Toners are in Appendix D.

### 5.6. Mixing Time Correlation And Validation

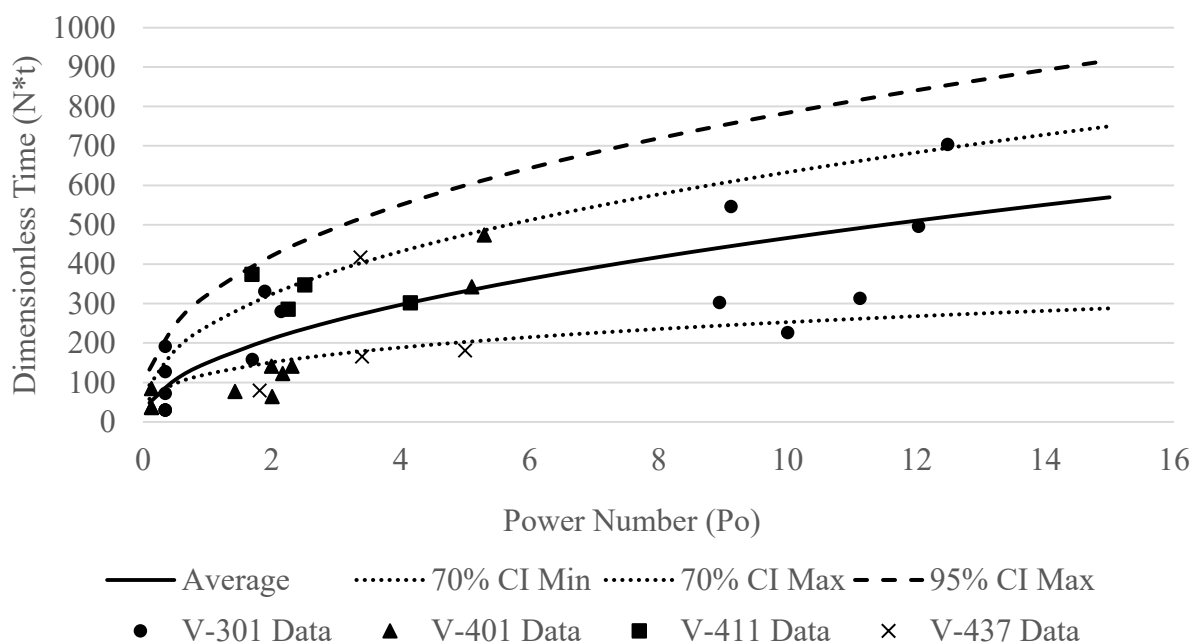
Once the mixing time correlation was established, curves were developed for the time it will take to reach 95% homogeneity at a specific viscosity and rotational speed. An example of these curves for the largest tank (V-301) is shown in Figure 12. Additional Mixing Time graphs are in Appendix E.





**Figure 12.** Mixing time curve at different viscosities and rotational speeds for V-301.

Once the relationship between power number and dimensionless mixing time was established, the collected data was graphed against the confidence intervals of this relationship (Figure 13). This shows that all the data falls within the appropriate confidence intervals. No data was collected for V-302 or V-421 because there were no products made on these tanks that had a defined mixing step post acid/peroxide addition.



**Figure 13.** Mixing time data graphed against the confidence intervals of the correlation.

To validate the model, all three tank null hypotheses needed to be rejected. This means that the variation the mixing time estimates comes from the mixing time measurements instead of the agitator power number equations. After taking the right tailed t-test, the p-values were 0.001, 0.013, and 0.038 for V-401, V-411, and V-437 respectively. These values are less than the alpha value of 0.05 which supports the constants used in the power number model.

## **6. Conclusion And Impact**

The purpose of this project was to create a systematic way to scale between mixing tanks. The method relies on the knowledge of the product viscosity at the process temperatures and the effect of shear on the viscosity. A quick experiment to measure viscosity changes with temperatures and shear rates can be done when a new product introduced. The method also relies on a process be established an at least one tank so that it can be scaled. Then using this information, the agitation speed graphs can be used to convert the speeds between tanks and with that speed, find the estimated time. The shear rate graph can be used to convert between speeds of the homogenizers and the flow rate graphs to find the homogenization time to keep the number of turnovers constant. This will reduce the time it takes to introduce new products and do transfers between tanks. Also, by knowing appropriate parameters that correlate between each of the tanks the number of reworked batches should be reduced. Overall, this project will save time and money as the company moves forward to expand their manufacturing capacity.

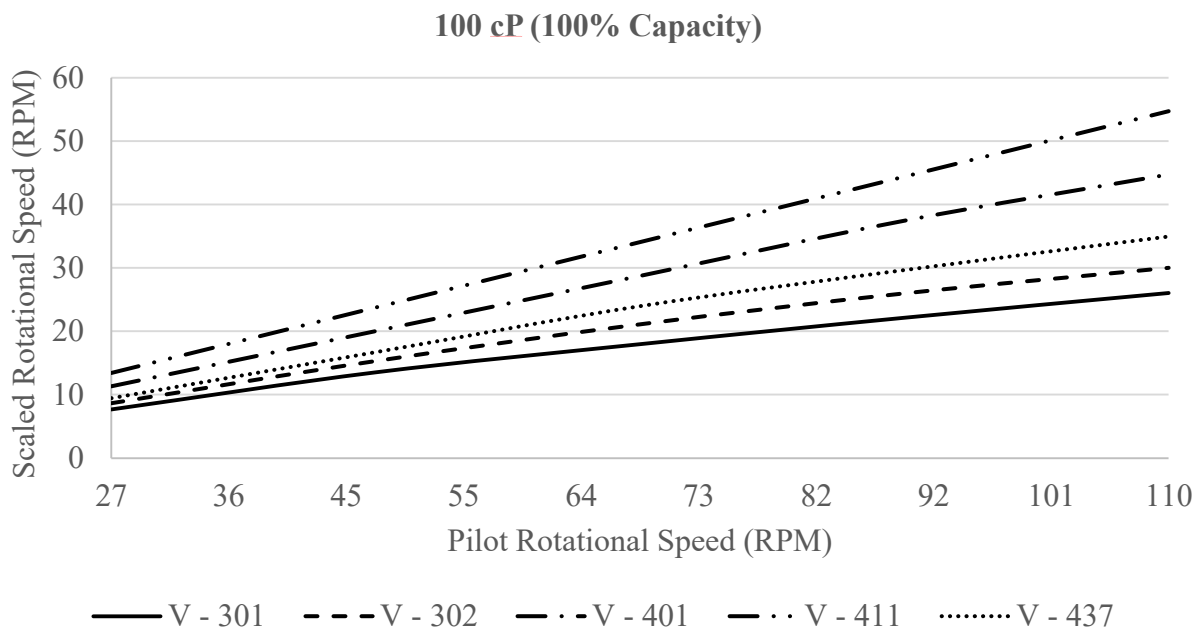
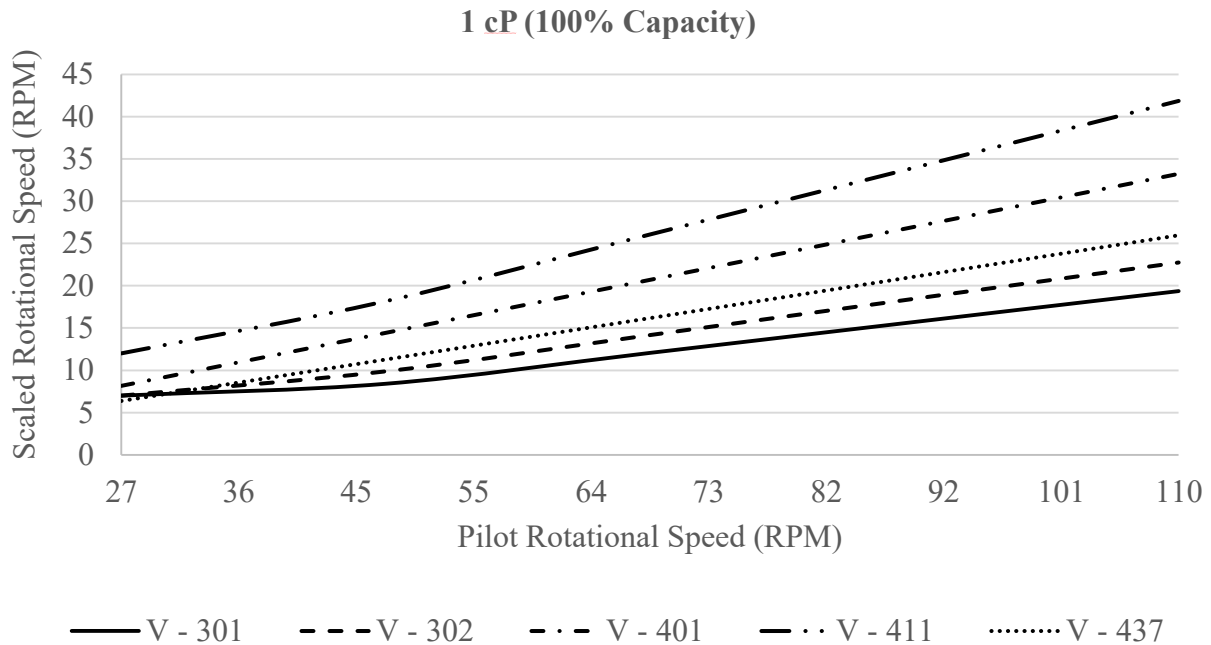
## 7. References

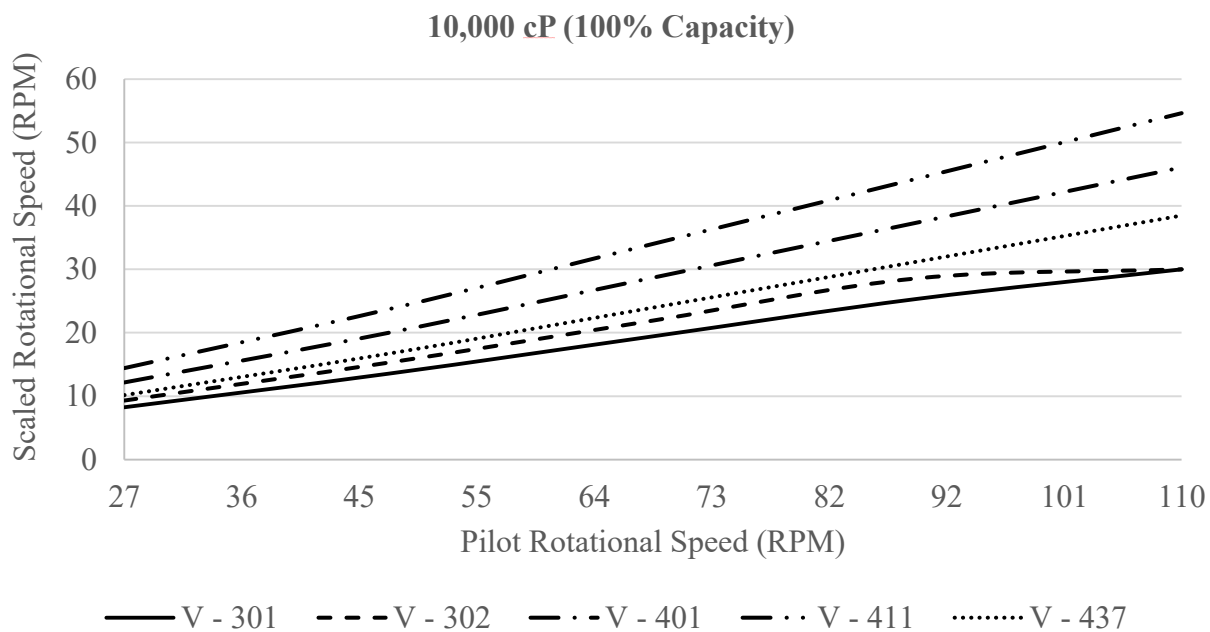
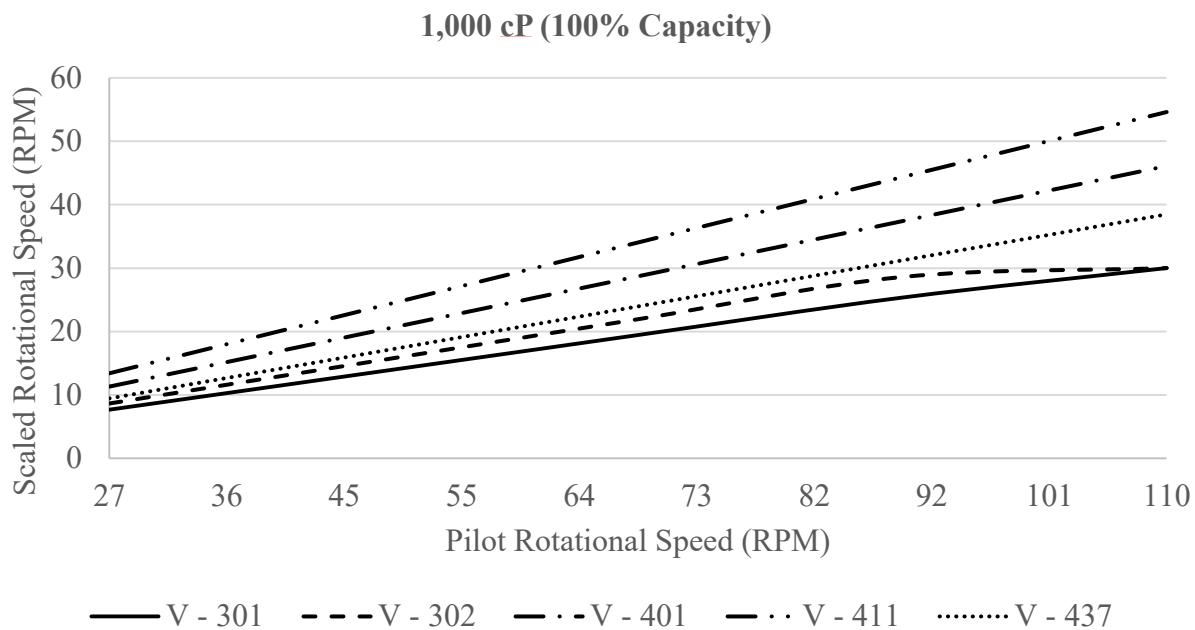
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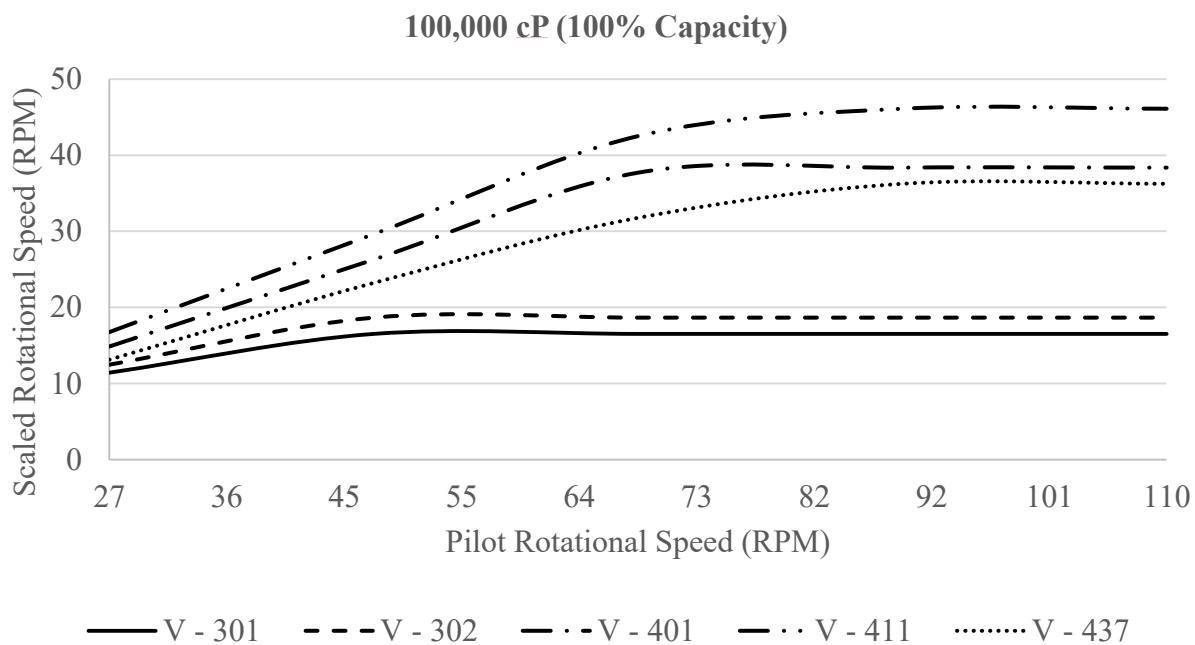
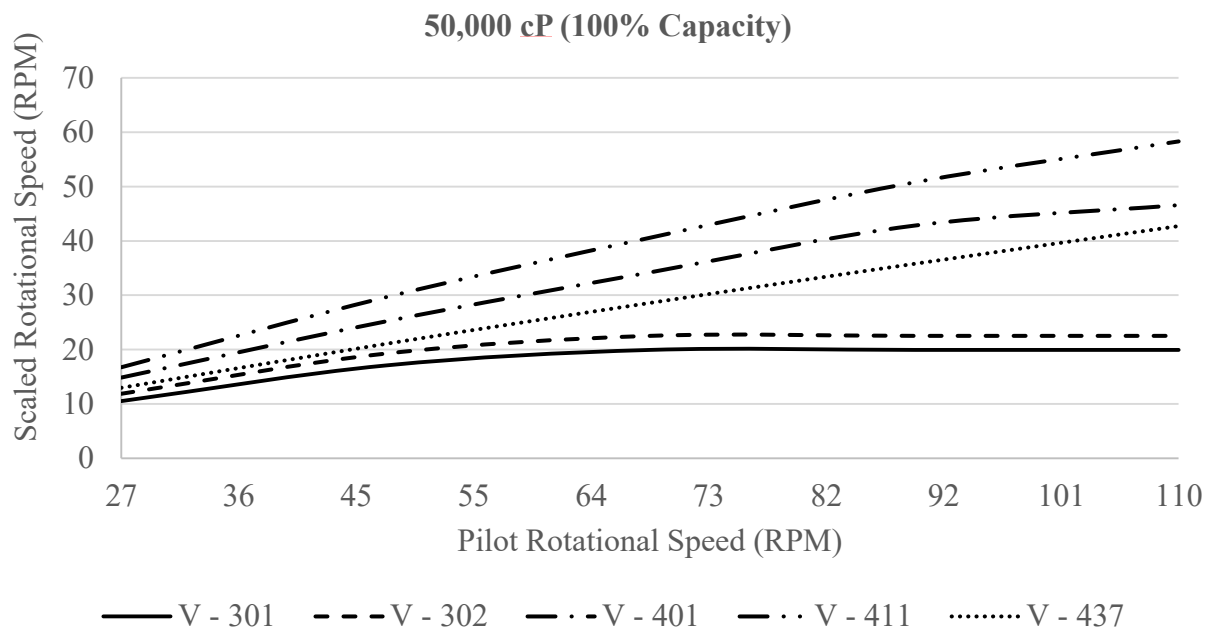
## 8. Appendices

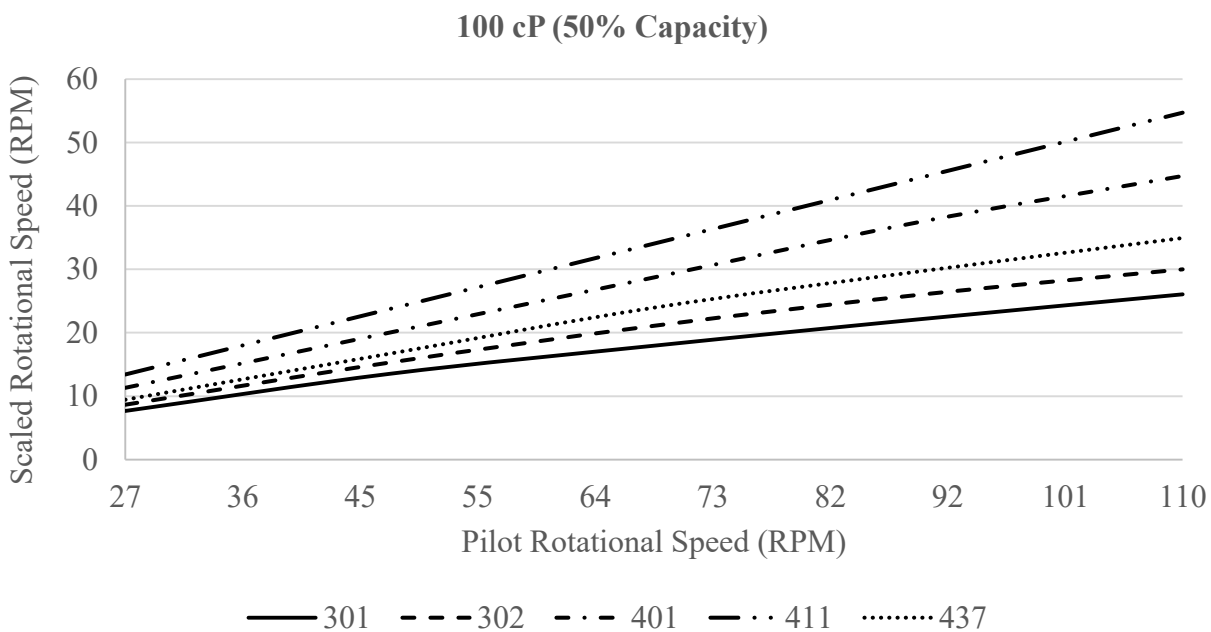
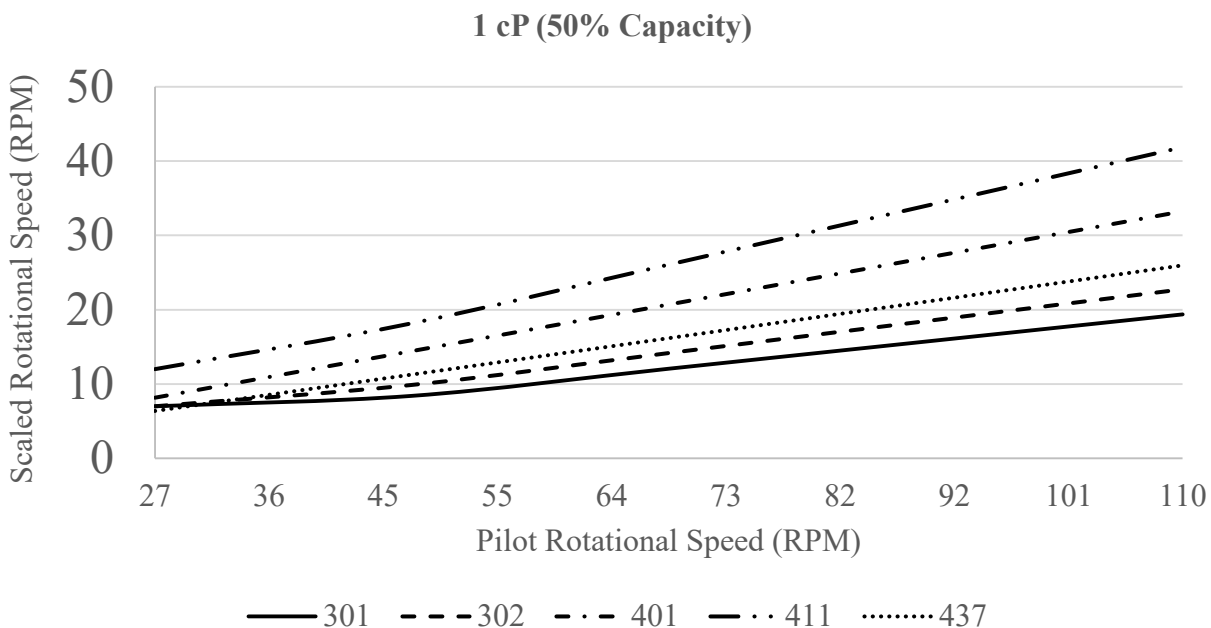
### 8.1. Appendix A

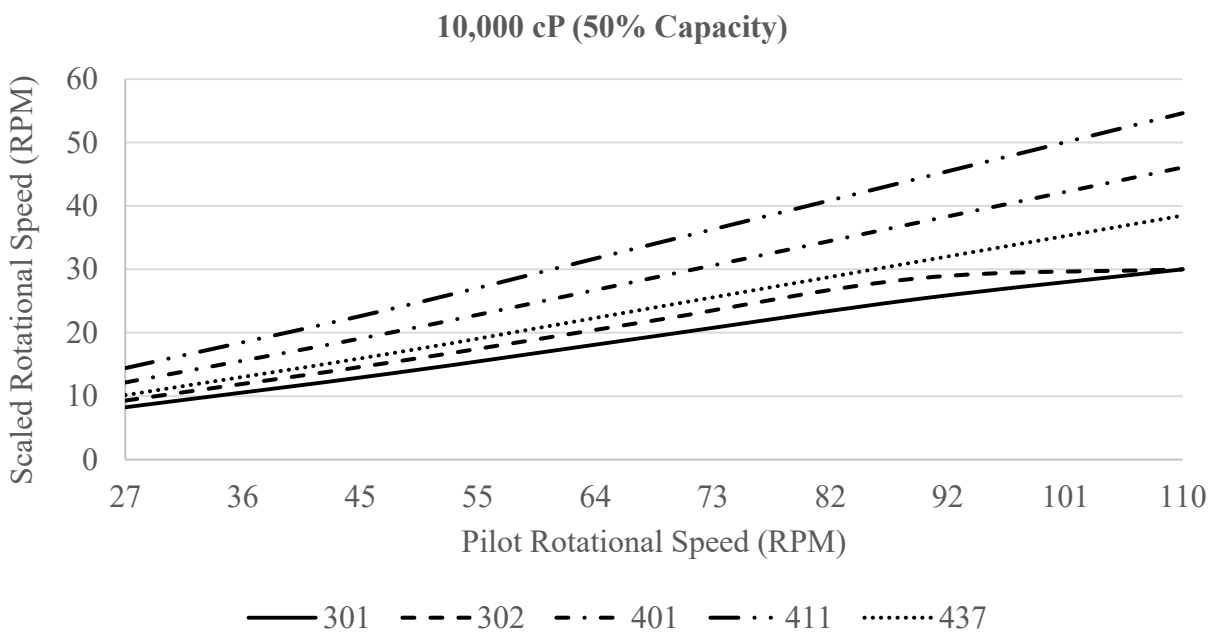
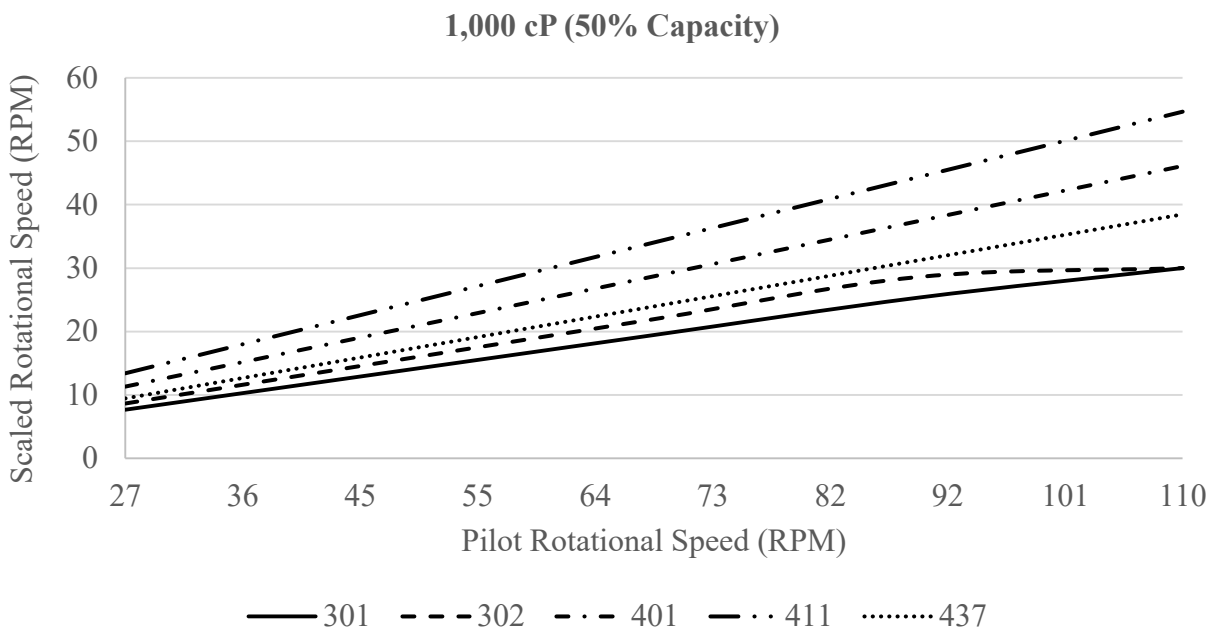
Graphs show mixing speed conversions for the tank agitators at a certain viscosity and percentage capacity (indicated in title).



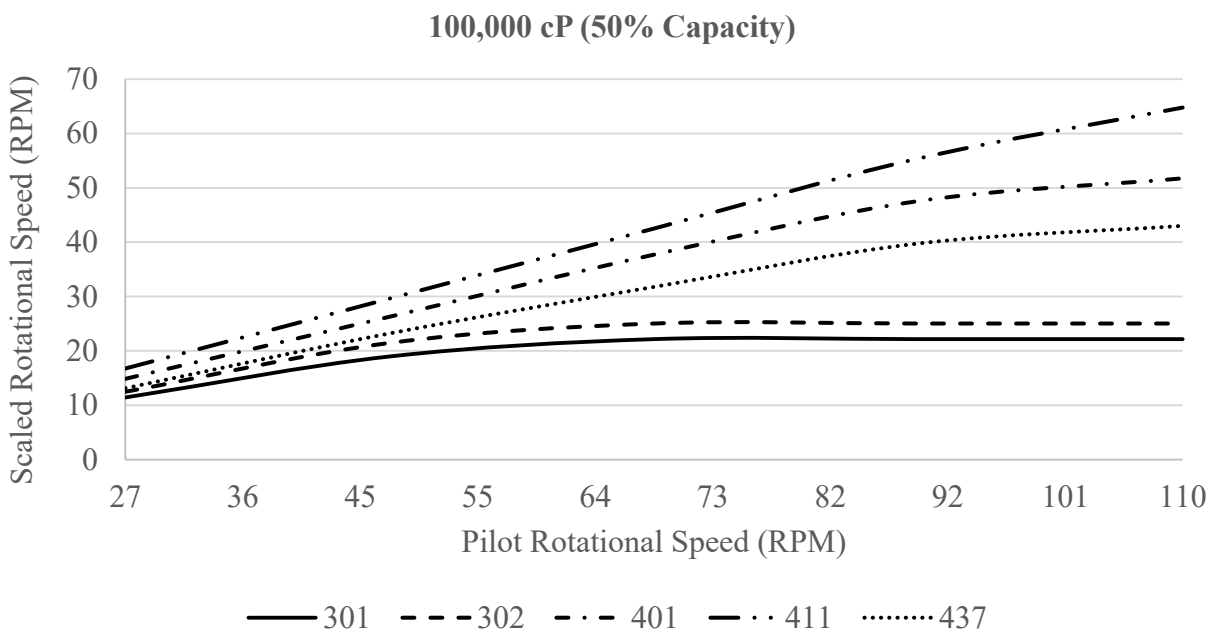
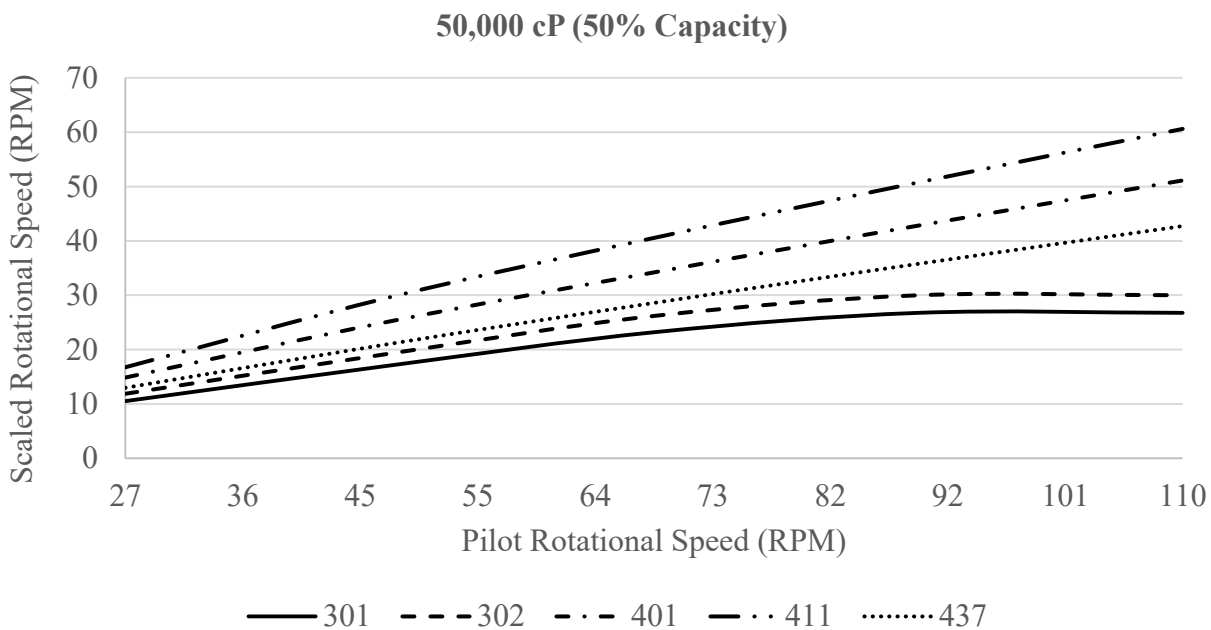






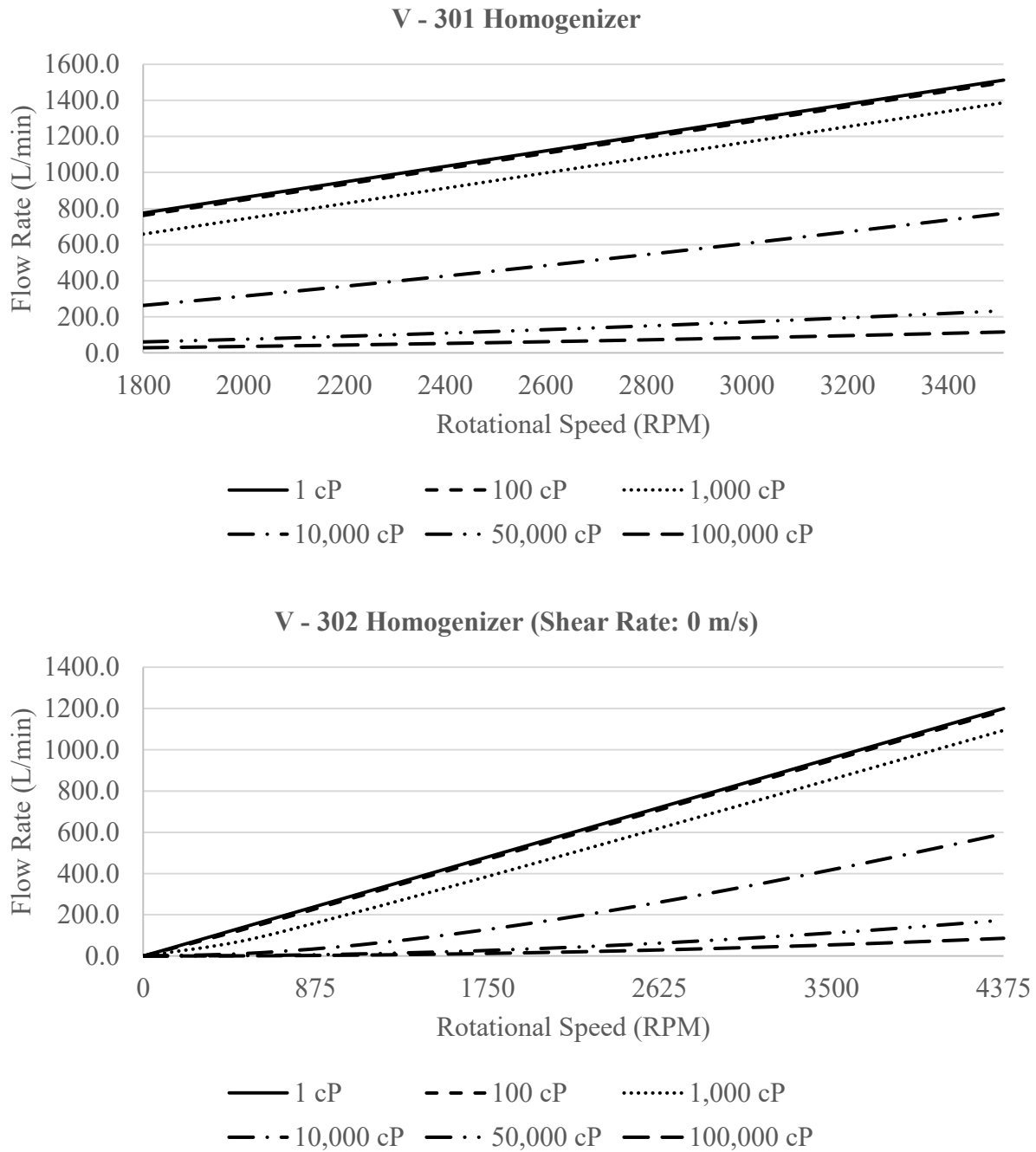




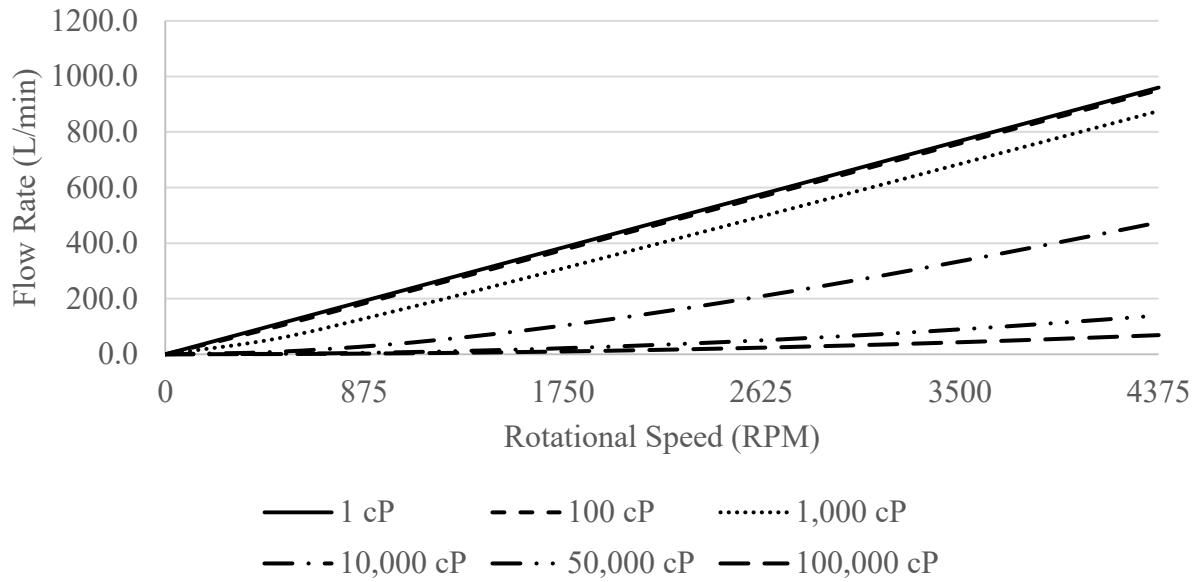


## 8.2. Appendix B

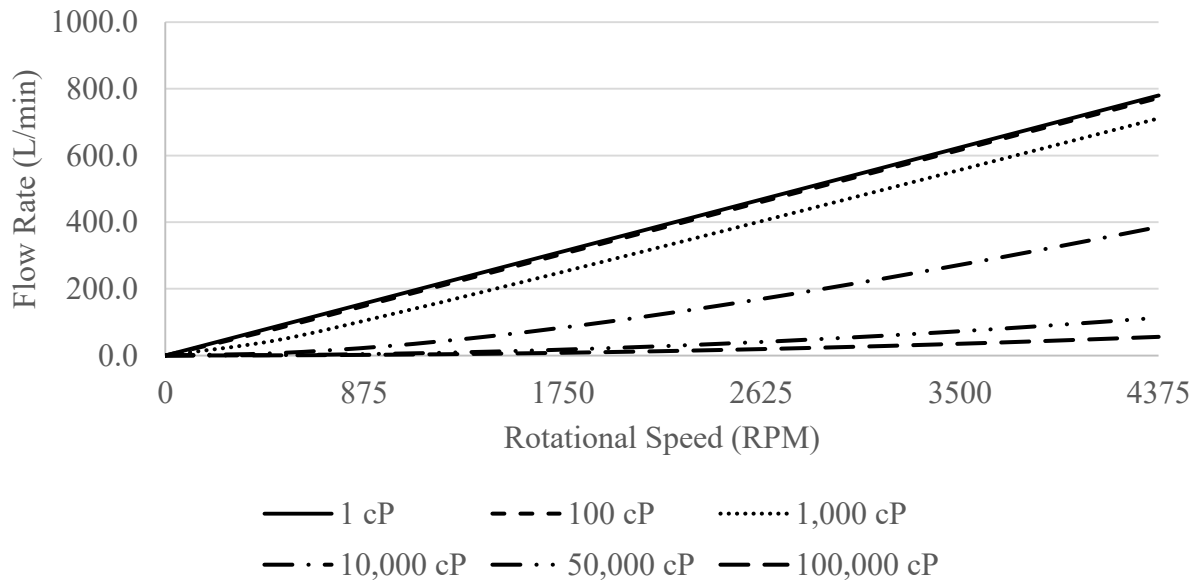
Graphs show the flow rate through the homogenizer on each tank with varying viscosity. The second largest tank (V-302) has a homogenizer with a moving stator that is characterized by shear rate. So a fixed stator for this homogenizer means a shear rate of zero even though it does have a shear rate because the rotor is moving.

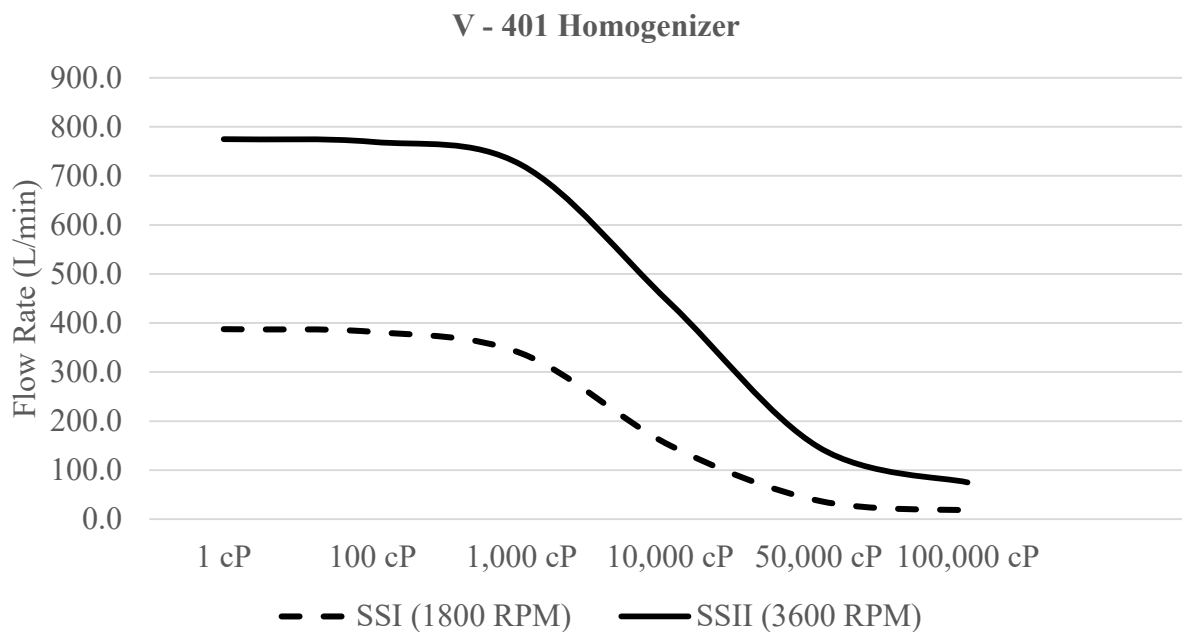


**V - 302 Homogenizer (Shear Rate: 30 m/s)**

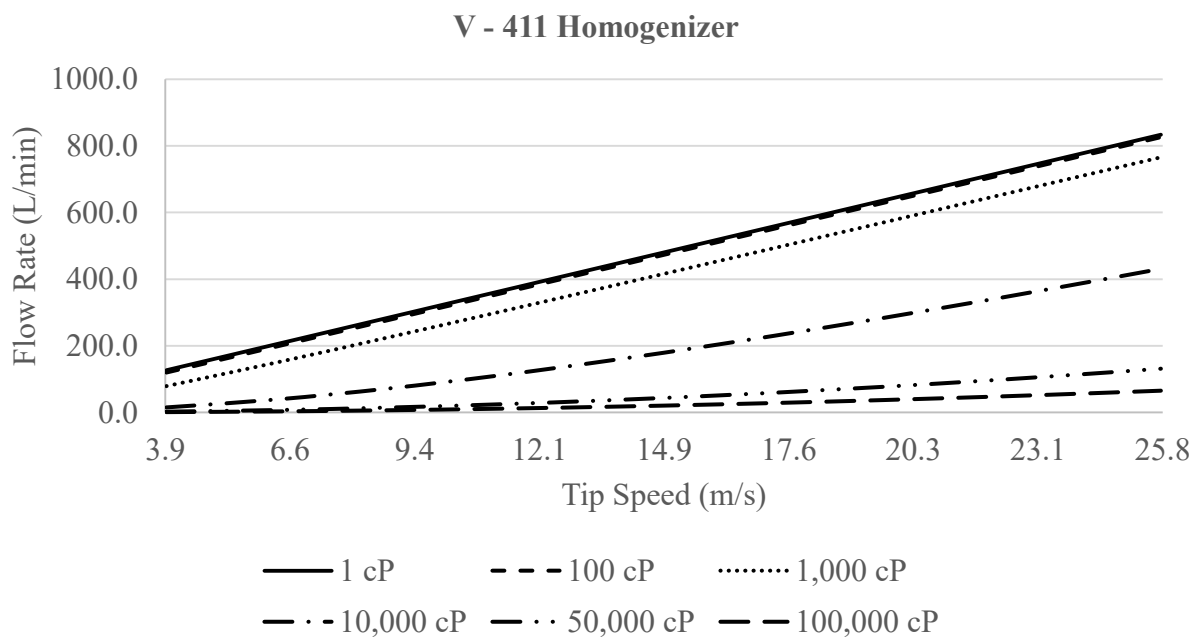


**V - 302 Homogenizer (Shear Rate: 60 m/s)**

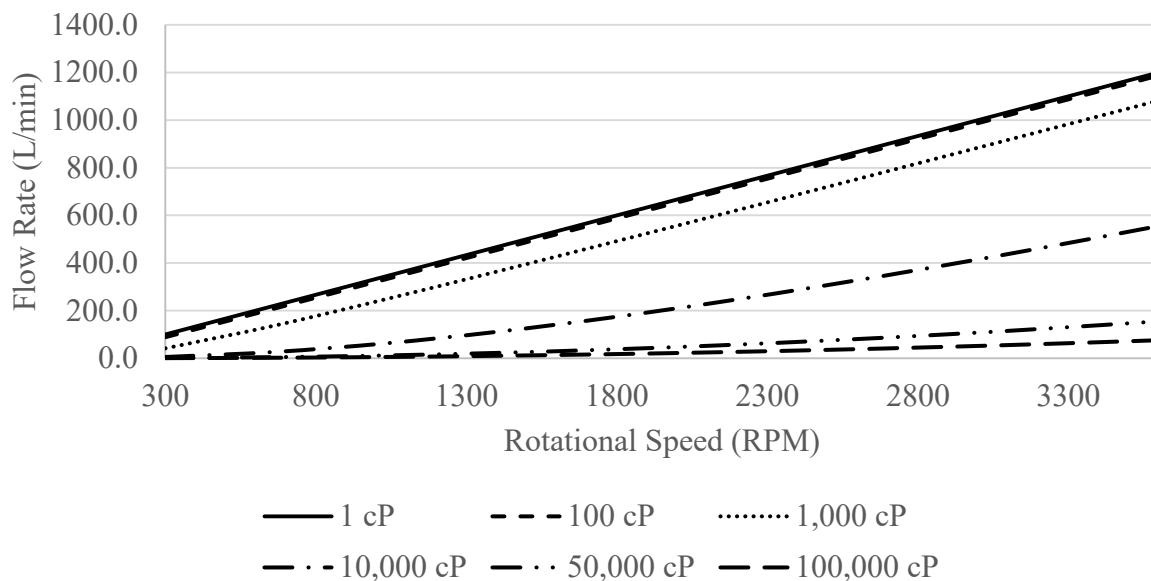




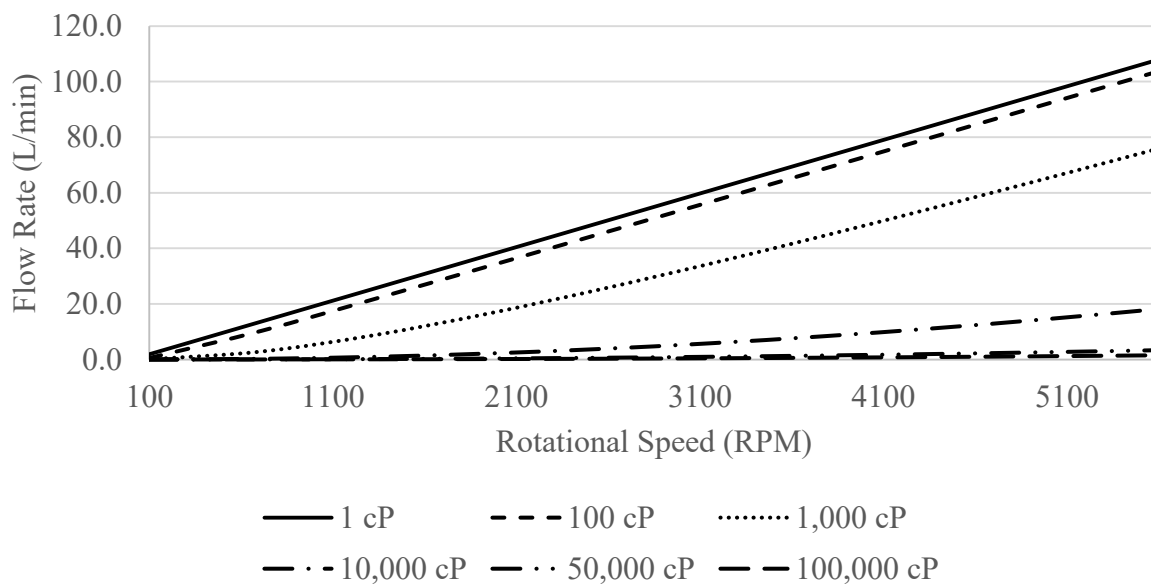
V-401 only have two speeds (SSI and SSII), so it's graphed against viscosity.



### V - 437 Homogenizer

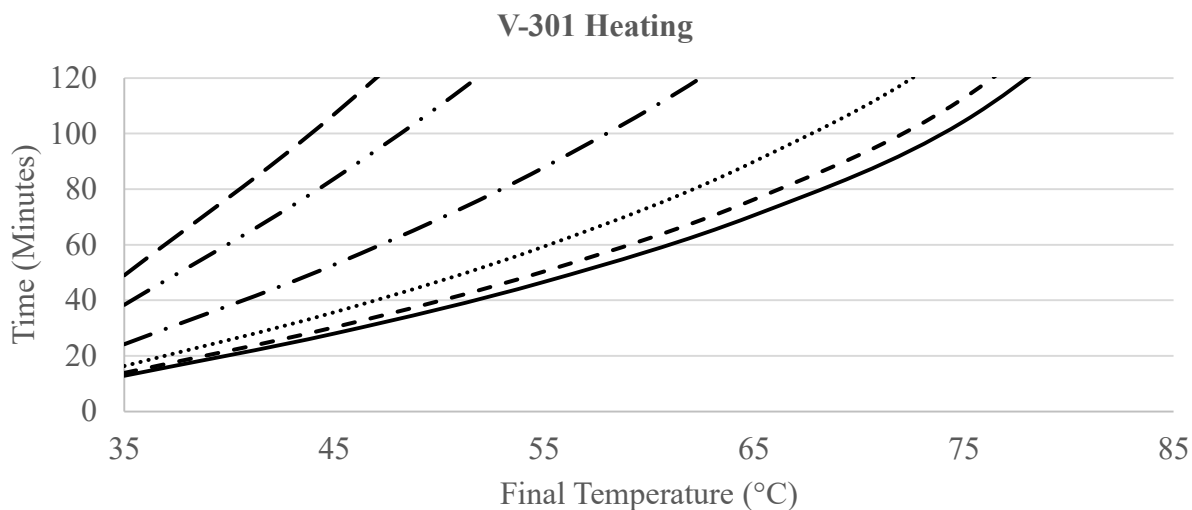


### V - 421 Homogenizer

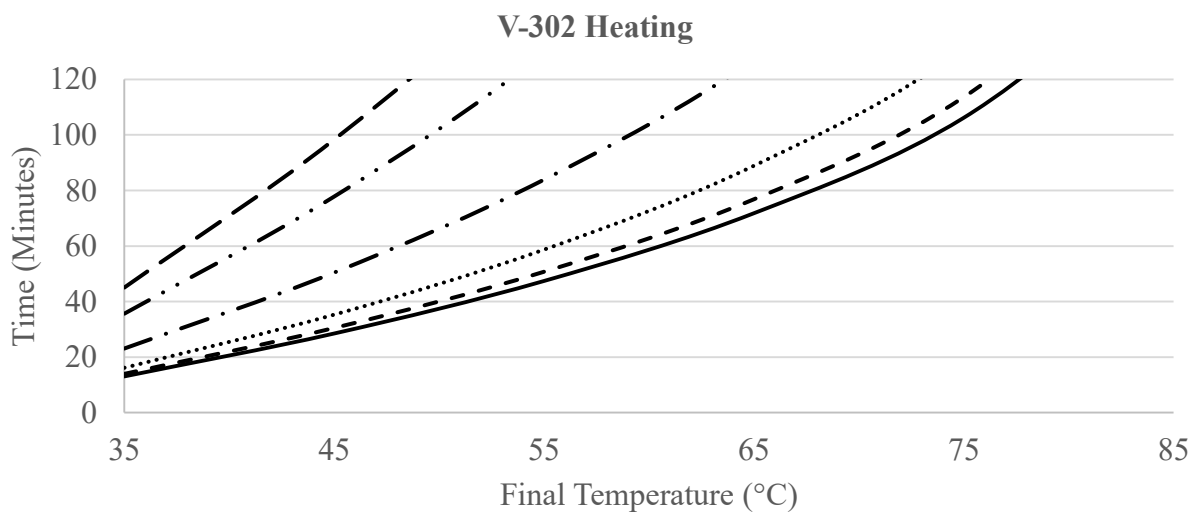


### 8.3. Appendix C

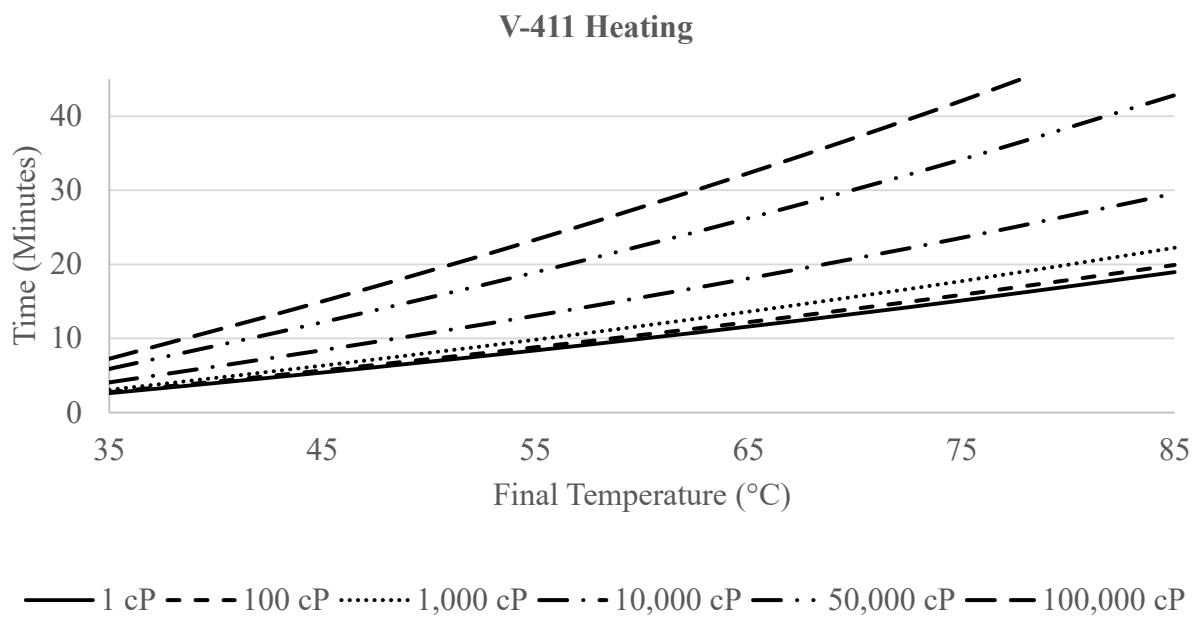
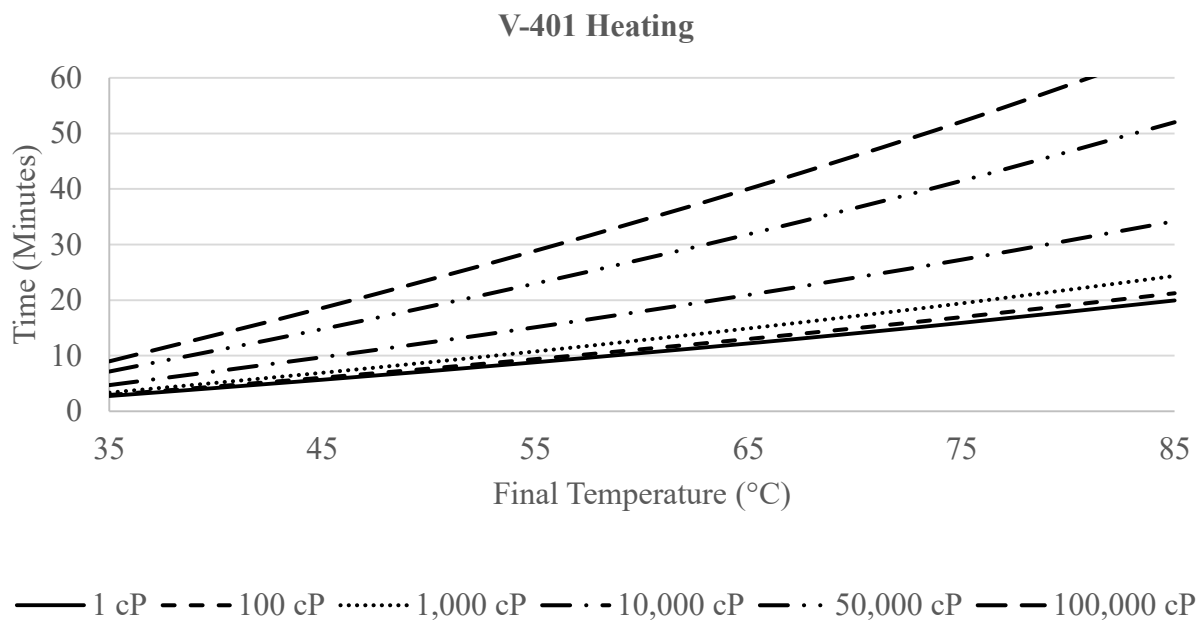
Graphs of heating/cooling times versus final/initial temperature at varying viscosities. All graphs are at a median agitator speed (V-301: 15 RPM, V-302: 15 RPM, V-401: 40 RPM, V-411: 40 RPM, V-437: 25 RPM, V-421: 40 RPM) and either an initial/final temperature of 25°C depending on heating/cooling.



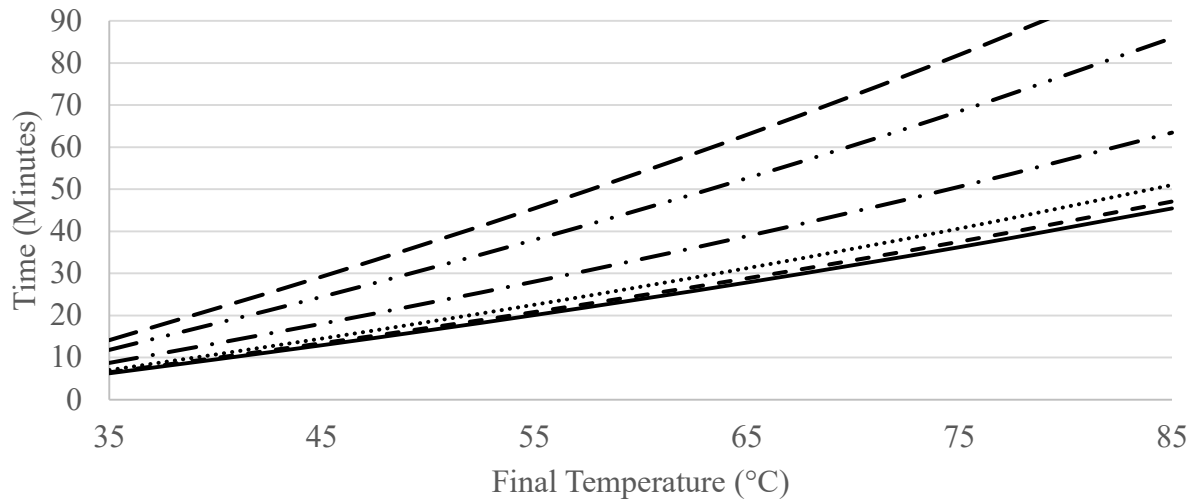
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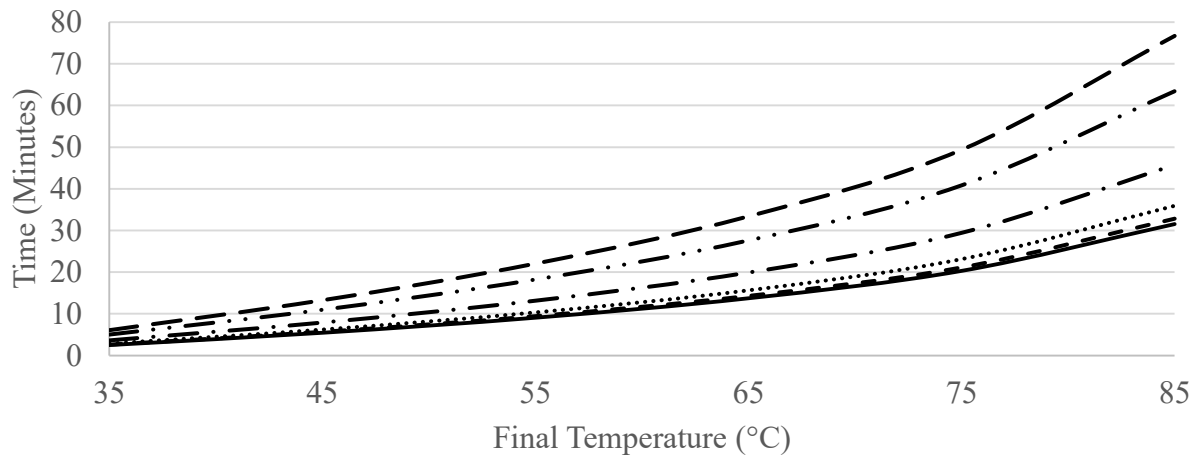


### V-437 Heating



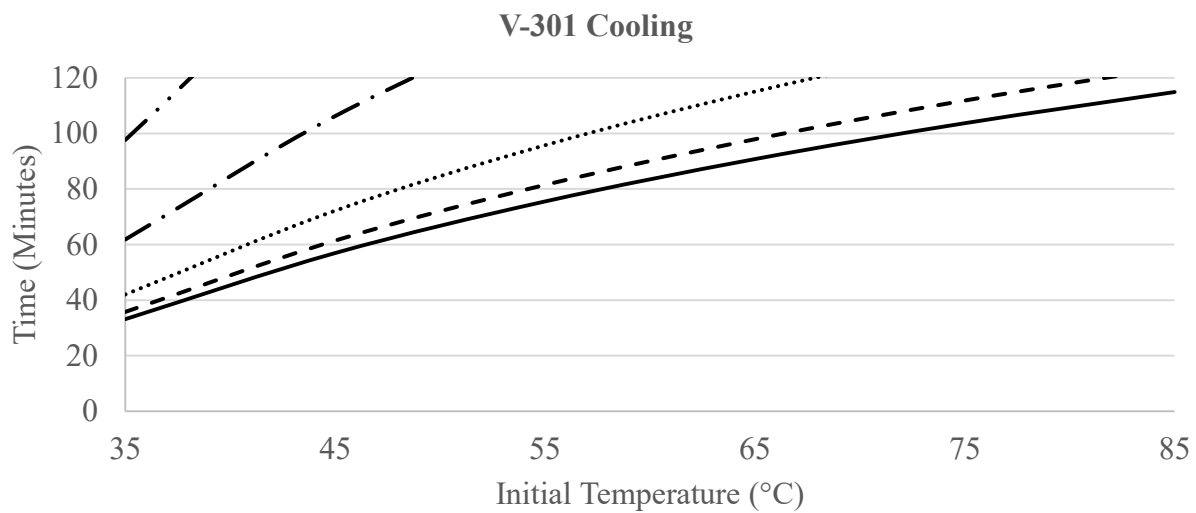
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### V-421 Heating

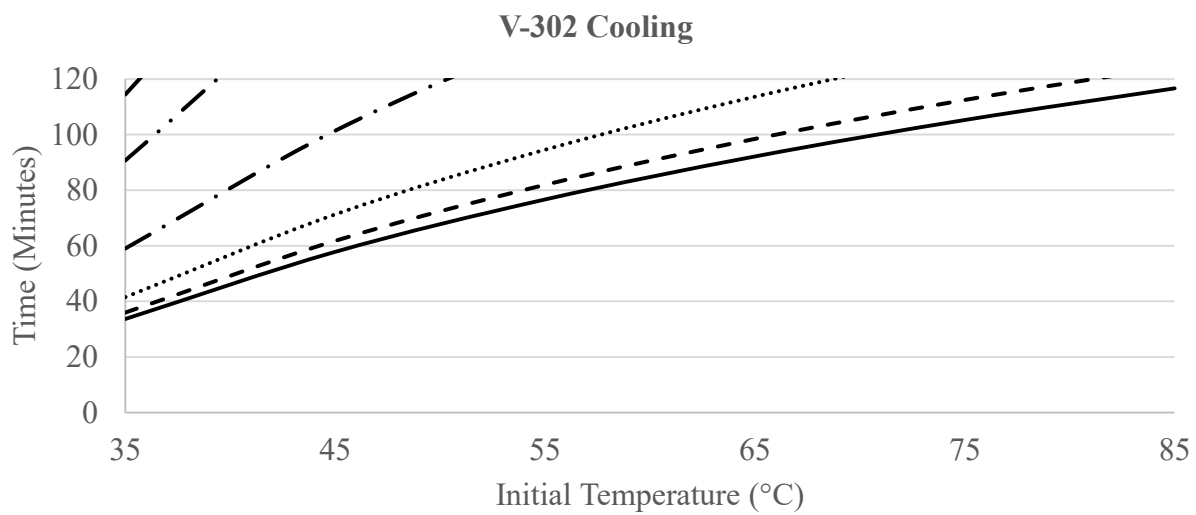


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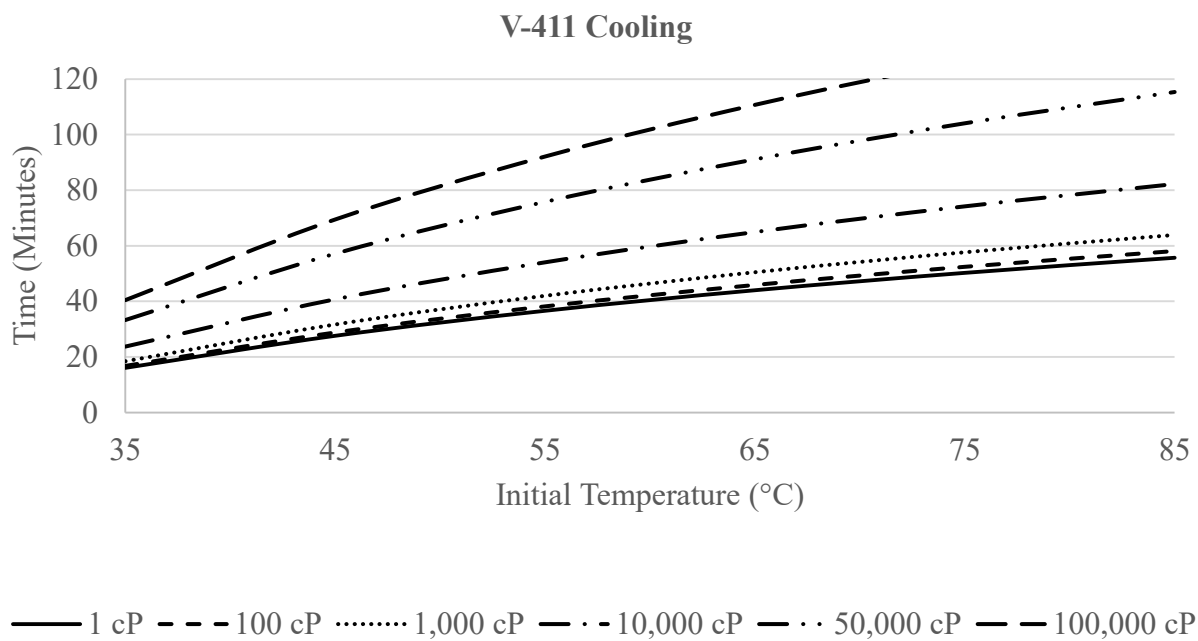
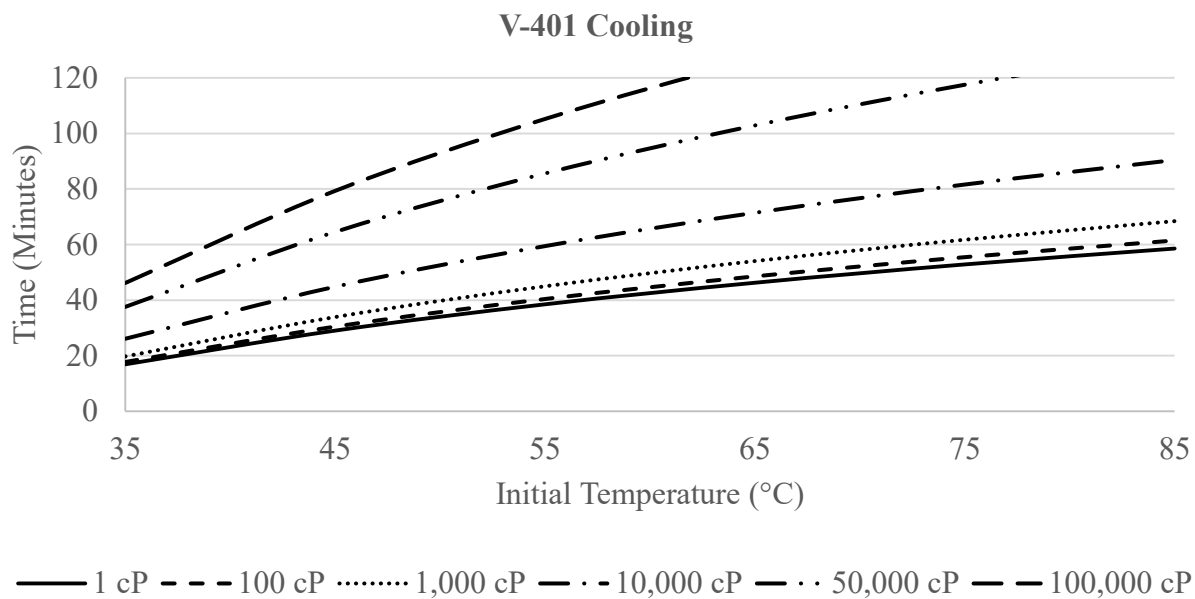


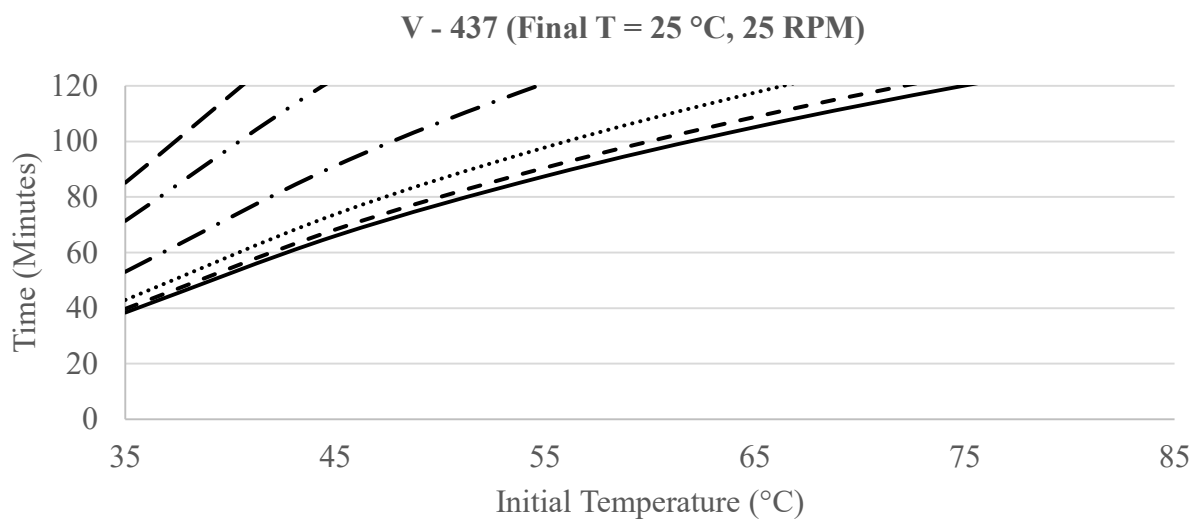


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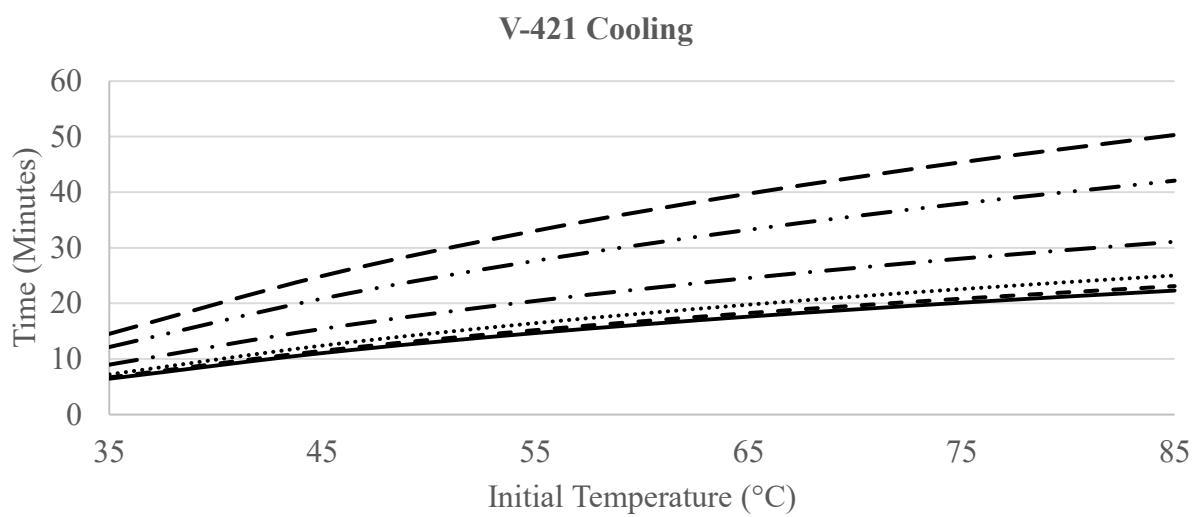


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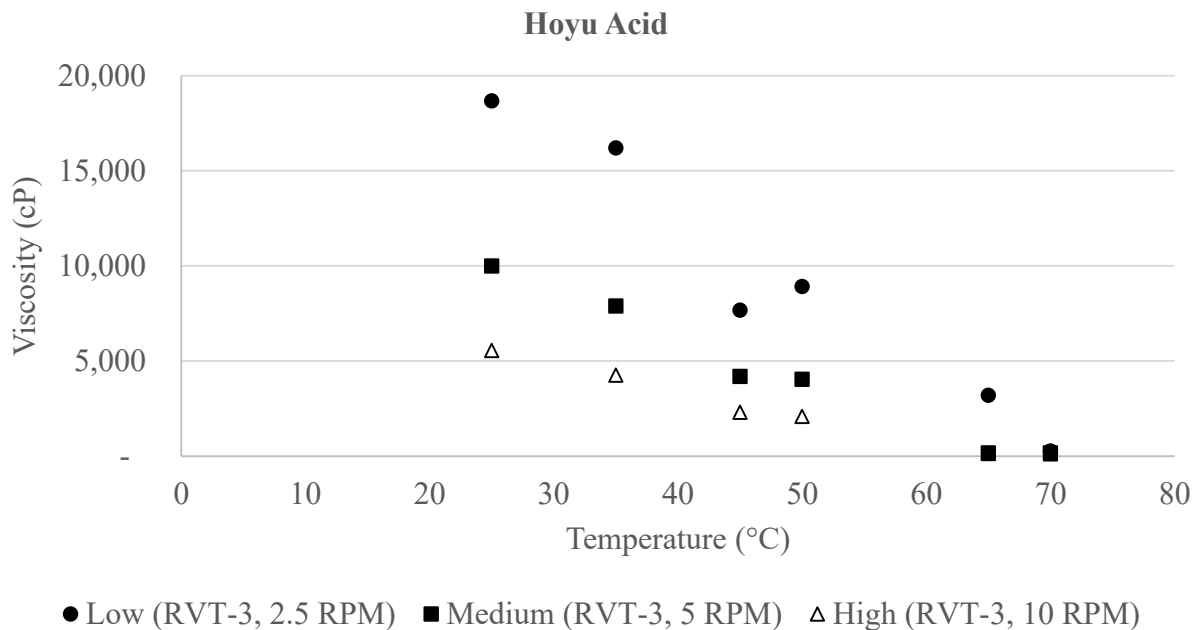
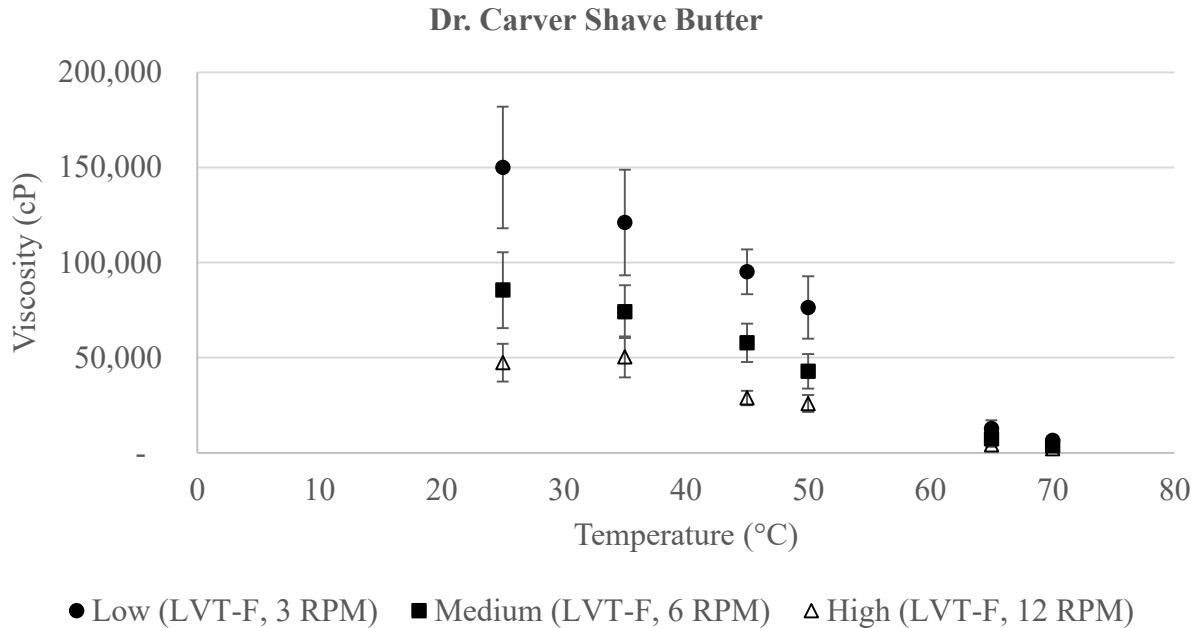
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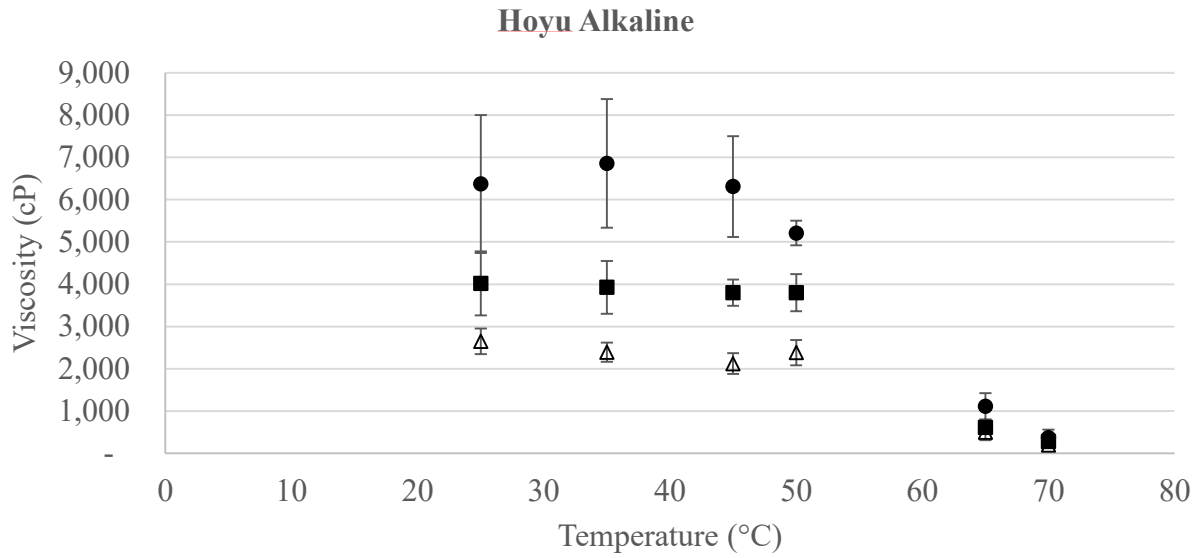
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#### 8.4. Appendix D

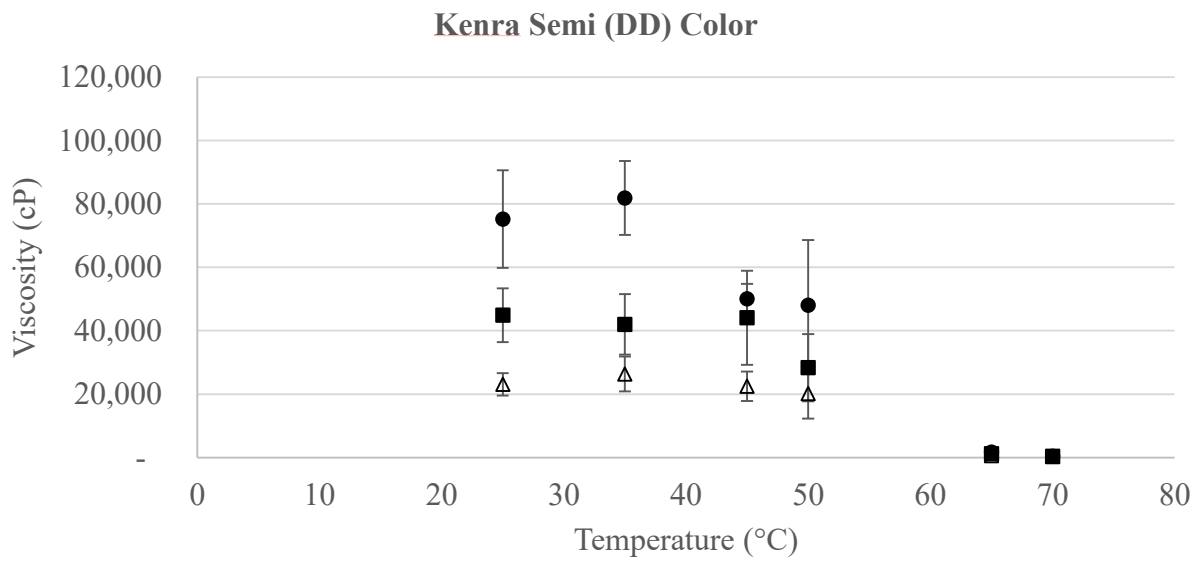
Graphs of viscosity versus temperature and shear. Medium shear rate is the parameter's C-Care uses for QA measurements in their Certificate Of Analysis. Thayer's Toner's were not measured because viscosity is that of water at all points in the process.



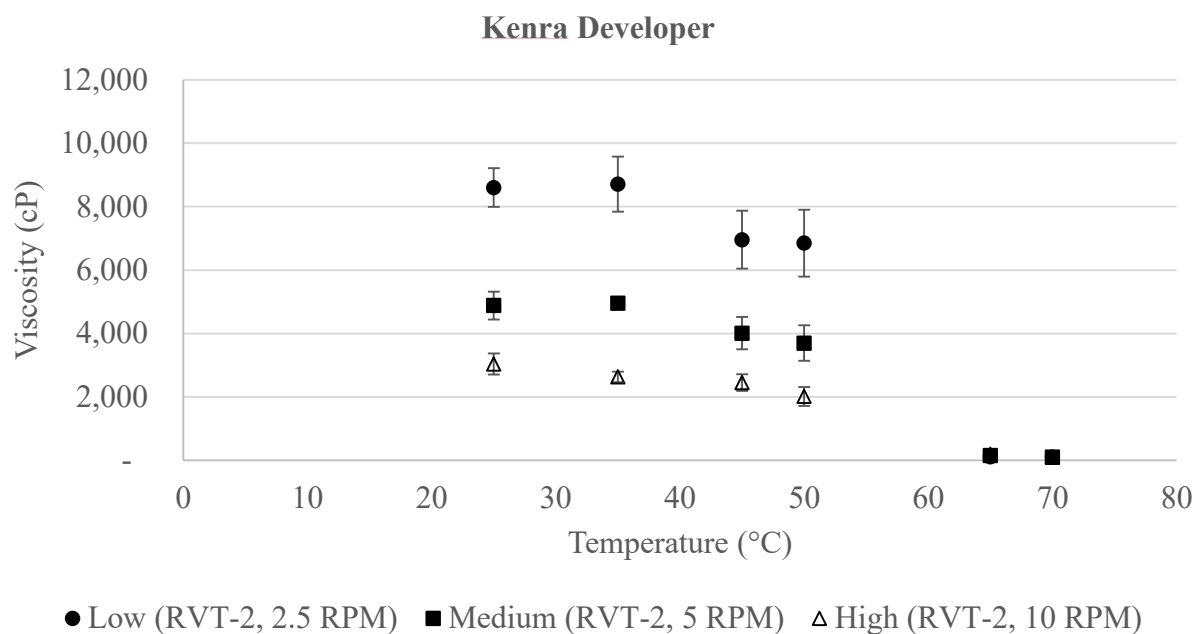
Hoyu acid was only taken one time because mixing time measured viscosities were less than 100 cP on all samples.



● Low (RVT-3, 5 RPM) ■ Medium (RVT-3, 10 RPM) △ High (RVT-3, 20 RPM)



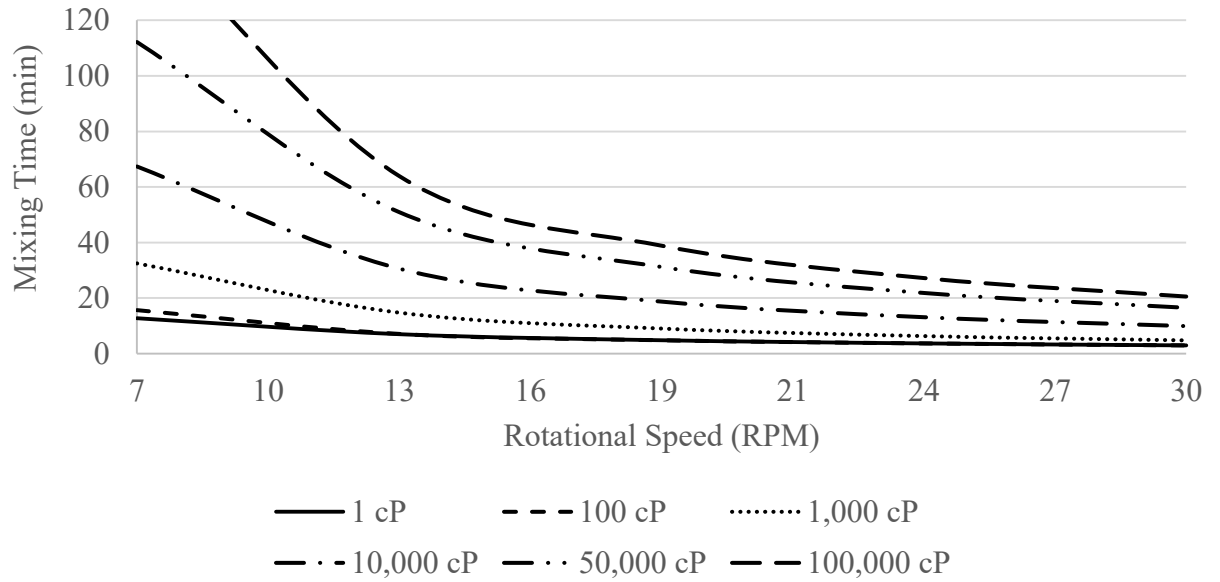
● Low (RVT-C, 2.5 RPM) ■ Medium (RVT-C, 5 RPM) △ High (RVT-C, 10 RPM)



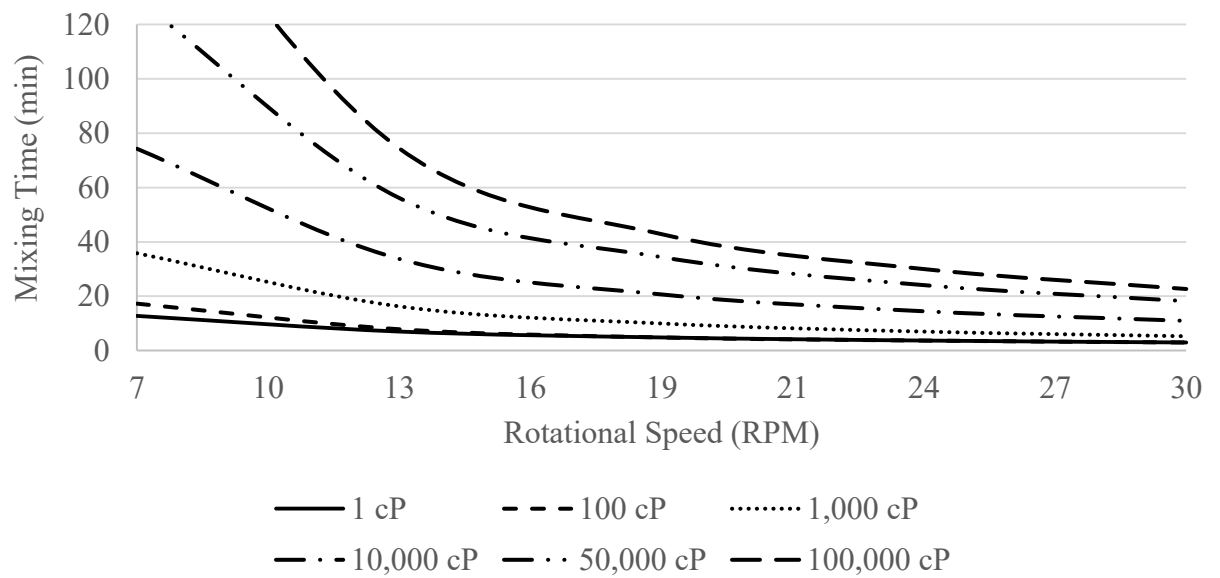
## 8.5. Appendix E

Additional mixing time graphs for each tank at different viscosities and agitator speeds.

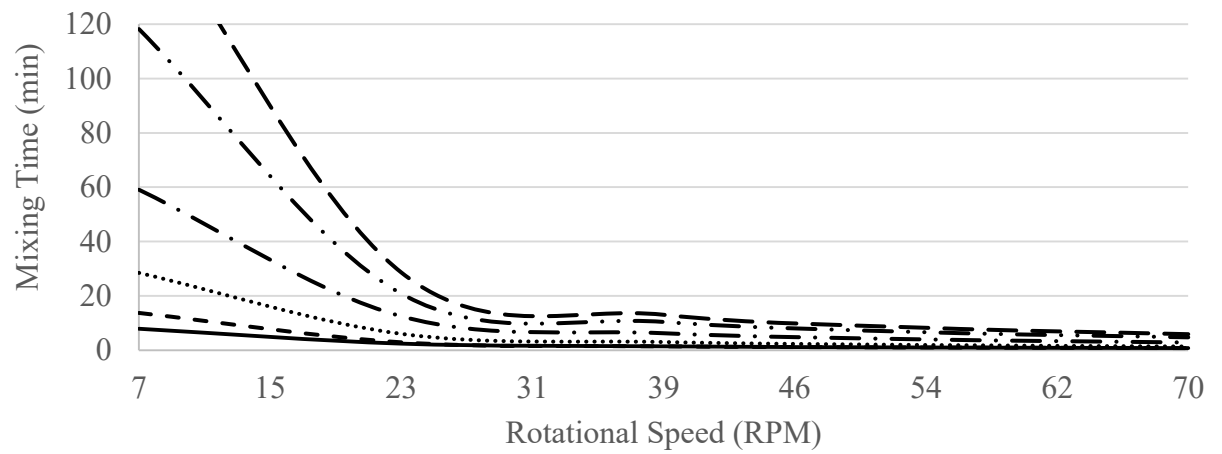
**V - 301**



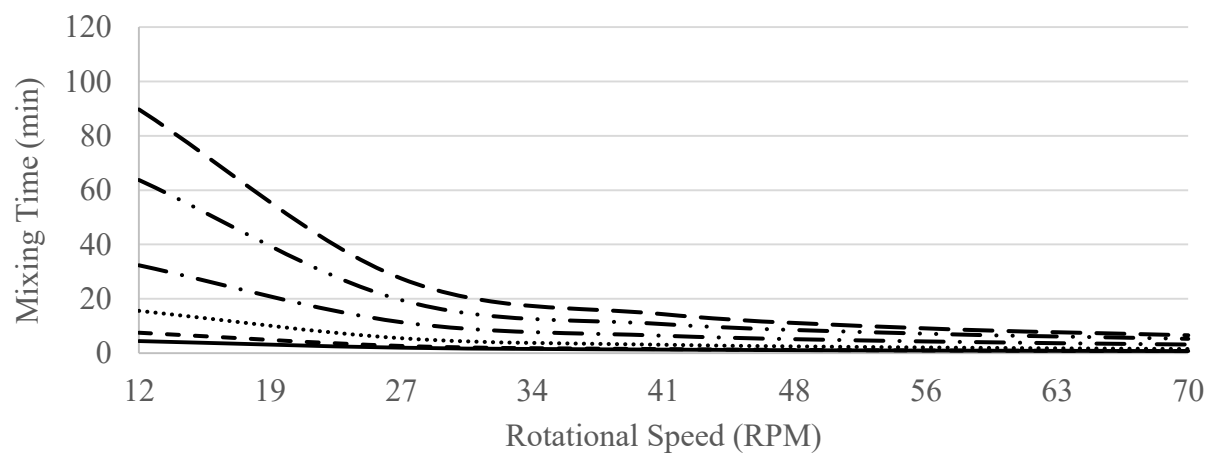
**V-302**



### V - 401

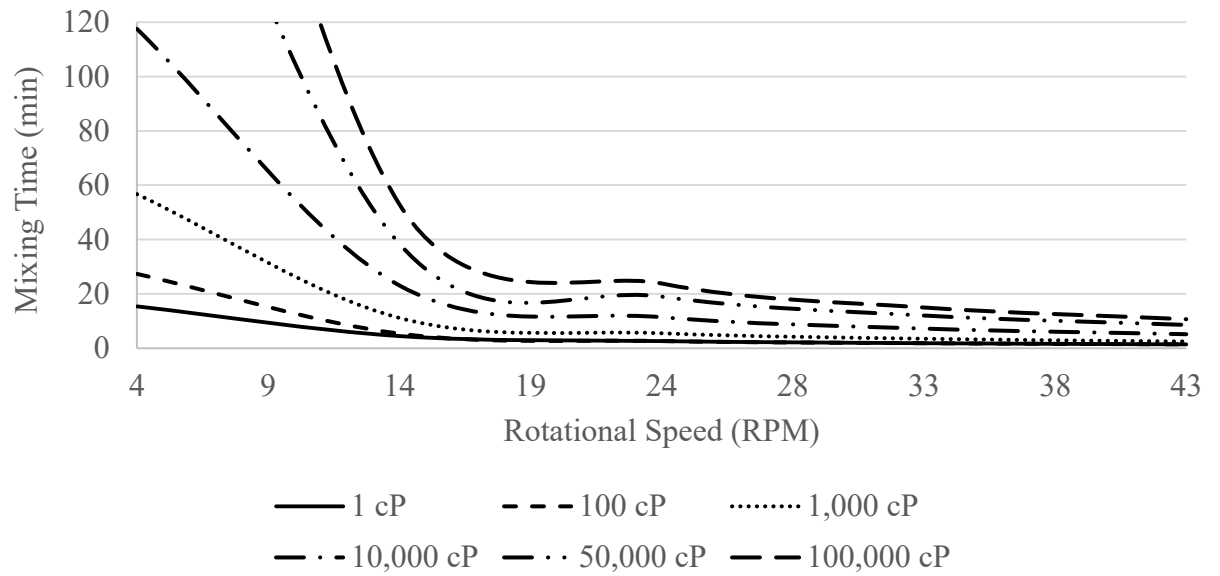


### V - 411

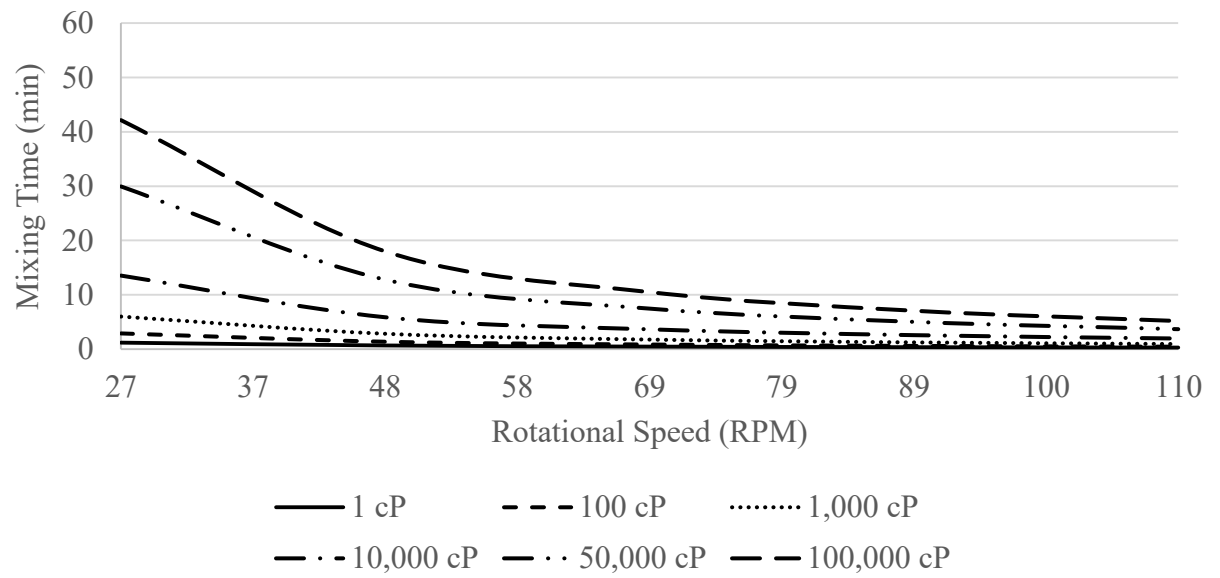




### V - 437



### V - 421



## 8.6. Appendix F

The table below shows specifications for the agitators and homogenizers on each tank.

Make	Agitator				Rotor-Stator Homogenizer					
	Drive Capacity	Rotational Speed (Min)	Rotational Speed (Max)	Diameter	Drive Capacity	Rotational Speed (Min)	Rotational Speed (Max)	Rotor Diameter	Shear Gap	Max Flow Rate
APV Crepaco	29.8 kW	7 RPM	30 RPM	2.13 m	22.3 kW	1800 RPM	3510 RPM	0.151 m	0.254 mm	25.20 L/s
APV Crepaco	22.4 kW	7 RPM	30 RPM	1.83 m	30.0 kW	0 RPM	4375 RPM	0.131 m	0.500 mm	20.00 L/s
Becomix	11.0 kW	7 RPM	70 RPM	1.24 m	23.0 kW	1800 RPM	3600 RPM	0.167 m	1.250 mm	12.92 L/s
Becomix	7.5 kW	12 RPM	70 RPM	0.99 m	23.0 kW	450 RPM	2955 RPM	0.167 m	1.250 mm	13.89 L/s
Symex	29.0 kW	4 RPM	43 RPM	1.58 m	23.0 kW	300 RPM	3600 RPM	0.136 m	0.500 mm	20.00 L/s
Becomix	0.6 kW	27 RPM	110 RPM	0.37 m	2.2 kW	100 RPM	5600 RPM	0.056 m	0.750 mm	1.80 L/s

## Thomas J. Plegue

tplegue@gmail.com

### SKILLS

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Project Management, Process Development, Biotechnology, Research, Development, Statistics, cGMP Manufacturing, Validation, Cell Culture, Safety, Experimental Design, Data Analysis

### WORK EXPERIENCE

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#### Continuous Improvement Engineer

**January 2018 – June 2018**

*C-Care, LLC.*

*Linthicum Heights, MD*

- Developed a systematic method for scale-up of mixers for faster process development.
- Managed data collection with operations team, in a dynamic CMO environment, complying with Good Manufacturing Practices (GMPs).
- Supported new product technology transfer and equipment using mixing knowledge.
- Reduced a product's batch time resulting in a \$200/batch (~\$12K/yr) savings.
- Updated and digitized P&IDs for further process improvements.
- Optimized QA process to reduce time using statistics; potential \$80K/yr savings.
- Led a research project to create new formulations for water soluble packaging.
- Recommended process improvements to reduce manufacturing costs and improve validation methods.

#### Clinical Engineering Research Associate

**June 2016 – July 2017**

*VA Healthcare System*

*Ann Arbor, MI*

- Led an investigation of coatings to stop biofouling on blood contacting medical devices.
- Managed a multidisciplinary research project through collaborations, material inventory, and scheduling of experiments.
- Developed an in place coating process that reduced processing time by 70% and expensive chemical waste. Process would make scale-up for manufacturing simpler.
- Optimized fabrication protocols and SOPs for a microfluidic device that reduced device size by 230% with same efficiencies as larger devices
- Presented work at a global conference to medical device experts and researchers.
- Plegue, T.; et. al. "Stability of Polyethylene Glycol and Zwitterionic Surface Modifications in PDMS Microfluidic Flow Chambers" *Langmuir* **2018**, 34 (1), 492-502.

#### Undergraduate Research Assistant

**September 2015- October 2016**

*Dr. Sunitha Nagrath Laboratory - University of Michigan*

*Ann Arbor, MI*

- Assisted with optimization of a microfluidic device to capture tumor cells, through capture efficiency of immunostained cell lines.
- Performed lab work for bioprocessing experiments in mammalian cell culture and isolation to collect data for devices.
- Managed the development and writing of laboratory protocols/SOPs for new processes.

**Associate Scientist/Engineer (Intern)****May 2015 – August 2015***BASF Chemical Company**Wyandotte, MI*

- Managed and performed experiments to improve adhesion by 400% between inexpensive polyolefin substrates and thermoplastic polyurethane coatings to reduce material costs.
- Collaborated with scientists, technicians, and managers to run experiments and obtain necessary materials.
- Presented recommendations for future studies to site experts and managers to achieve consistent surface coating adhesion on a large scale based on lab test results.
- Wrote standard operating procedures (SOPs) for processes to enhance operator safety.
- Won a cross-functional team challenge. Developed concept, with technical and economic analysis, to improve use of sustainable energy on site. Potential \$50K/yr savings identified.

**Project Manager****September 2014 – April 2015***Multimedia Educational Laboratory - University of Michigan**Ann Arbor, MI*

- Spearheaded a webpage that catalogs and describes equipment Drug Substance manufacturing: handling, reactions, drying, and separations.
- Consulted with new developers to add additional types of Drug Product processing equipment.
- Managed the development of other general equipment pages on the overall site.

**LEADERSHIP EXPERIENCE**

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**Project Manager****September 2013 – April 2017***BLUELab Biogas Local – University of Michigan**Ann Arbor, MI*

- Increased team size by 500% and converted a failing project into a viable, \$30K funded project by forming partnerships, focusing team involvement, and providing a collaborative environment.
- Managed and mentored a cross-functional team of 20 people working to develop a bioreactor for food waste breakdown targeting waste reduction and energy production.

**Eagle Scout**

- Managed one of the largest projects (\$30K) in the district. Mentor and teach scouts about chemistry and engineering.

**PROJECT EXPERIENCE**

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**Chemical Engineering Process And Design**

- Team appointed manager developing a lidocaine plant concept based on an MIT research project. Team developed PFDs, P&IDs, equipment specs, capital costs, and demonstrated favorable economics.

**Project in Design: Pharmacokinetics**

- Created a distribution model for Genentech's Cotellic and Zelboraf melanoma drugs.

**Metabolic Systems Engineering**

- Created a cell culture model for E. Coli using metabolism profiling, mass oxygen transfer modeling, and cellular engineering principles. Learning about fermentation techniques.

## EDUCATION

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### **Johns Hopkins University**

*M.S.E in Chemical & Biomolecular Engineering, GPA: 3.53/4.00*

**Expected June 2018**

*Baltimore, MD*

### **University of Michigan**

*B.S.E in Chemical Engineering, Minor in International Engineering*

*Cum Laude Honors, GPA: 3.24/4.00*

**April 2017**

*Ann Arbor, MI*